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THE INSTITUTE OF PETROLEUM

AN Ordinary General Meeting of the Institute was held at Manson House, 26 Portland Place, London, W.1, at 5.30 p.m. on Wednesday, November 8, 1950, the President, Mr C. A. P. Southwell, M.C., in the Chair.

THE GENERAL SECRETARY (D. A. Hough) read the list of elections since the preceding Ordinary General Meeting.

THE CHAIRMAN: I now have the pleasant duty of introducing our lecturer to-night. Dr Myddleton has had a long experience on the chemical side of the oil industry, having started in earlier days as a lecturer in chemistry at London University; during recent years he has been engaged in the cosmetic industry.

The following paper was then read by Dr Myddleton:

THE STABILITY OF MINERAL OILS IN TOILET AND COSMETIC PREPARATIONS

By W. W. MYDDLETON (Fellow)

SUMMARY

Toilet and cosmetic preparations containing mineral oils deteriorate during storage in unfavourable conditions. Deterioration is in part due to the instability of the oils themselves.

In order to assess the stability characteristics, the oils and emulsions made from them were exposed to sunlight for four months and an order of deterioration established.

Stability was found to be well indicated by the levels of bromine substitution values, aniline points, and the nature of the fluorescence of the oils in ultraviolet light.

INTRODUCTION

MINERAL oils are used in a wide variety of toilet and cosmetic preparations, including face-creams, hand-creams and lotions, sun-tan preparations, lipsticks, shaving-creams, brilliantines, and hair-dressings, in the emulsified as well as the unemulsified states. For all these purposes the main valued characteristics of the oils are bland emollient action on the skin, combined with lubricating properties and the property of imparting desirable physical attributes to the preparations made from them.

The chemist concerned with the manufacture of these varied products selects the grade of oil to be used with a view to the odour, colour, consistency, and texture he desires to impart to the product. He is concerned above all to avoid using any material which may be under the slightest suspicion of producing undesirable dermatological action.

The white oils are normally chosen, because they are obtainable practically free from odour and colour and because long experience has shown them to be without harmful physiological action on the skin.

The mechanism of emulsion-making requires that the oil should be capable of spreading on a water surface, and this characteristic is generally desirable also to achieve efficiency in the uses for which the non-emulsified oils are intended. The necessary spreading can be imparted to the white oils by adding certain perfumes, and in the case of those oils required for emulsification, suitable emulsifying waxes or heavy metal salts to promote and stabilize emulsification.

The employment of mineral oils gives rise to important problems concerned with the stability of the products containing them, and this is to some extent determined by the stability of the oils.

Toilet and cosmetic preparations sometimes receive extraordinary treatment before and after they reach the consumer. Shop-window displays, frequently in full sunshine, put the articles to a very stringent test. Practical trials have shown that the temperature of an emulsion in a glass bottle placed in a shop window facing south can reach and retain a temperature of 62·5° C for about 1 hour in the middle of a bright summer day.

The consumer frequently places a bottle he is using on a window-sill or shelf exposed to sunlight, and here the conditions may be worse than in the shop window. The air in the bottle is renewed once or twice daily, and the volume of air increases from day to day as the product is used.

Under the conditions described, the mineral oils themselves undergo undesirable changes, and the rapidity with which they occur and the extent to which they proceed is influenced, sometimes in the desirable, sometimes in the undesirable direction, according to the nature of the perfume or emulsifier added. This complication is under investigation, but in the meantime it is desirable to publish some observations made on a number of different oils without additions, and on emulsions of the same oils made with addition of an emulsifying wax which at the same time acts as a very mild oxidation inhibitor.

The undesirable characteristics developed in a toilet product containing mineral oil during exposure to light and heat and contact with air are a change in odour, usually of an unpleasant turpentiny nature, a darkening in colour, sedimentation in clear oils, and a breakdown of emulsions.

Although additions such as perfumes may play an important part in these changes, it is desirable to aim at a quality of oil most resistant to the type of change described. The chemist concerned in the industry requires rapid methods of testing to enable him to select from amongst a number of available oils those which will give the greatest protection against deterioration of his products, and to carry out routine checks on deliveries.

Experimental Procedure

As a basis for deciding on the type of test most likely to be generally useful, a number of technical white and half-white oils, enumerated in Table I, have been examined by exposing to sunlight in half-filled white glass jars over a period of several months. The oils were then assigned an order of deterioration with reference to odour, colour, and sedimentation. Control samples were kept in the dark.

Most of the oils were exposed in an unshaded window facing south, and a month later four were placed in a window facing west.

TABLE I

Oil no.	Sp. gr. 60° F/60° F	Redwood No. I Viscosity at 70° F, sec	Aniline point ° C
1	0·850	91	97·4
2	0·855	120	99·7
3	0·856	122	99·2
4	0·864	125	95·6
5	0·867	127	91·8
6	0·868	139	95·7
7	0·871	122	92·8
8	0·872	154	95·8
9	0·875	135	91·8
10 *	0·875	139	90·0
11	0·879	224	95·9
12 †	0·883	101	80·0
13 †	0·882	102	79·8

* Faint yellow. † Pale yellow.

All the oils developed a turpentiny odour, and the oils which developed it most quickly became most pungent at the end of the period, and showed a yellowing keeping pace with the odour. The formation of an orange-coloured sludge was observed in half-white oils, and slight turbidity was noted in most of the white oils, with a white sediment in one (see Table II).

TABLE II

Condition of Oils after Four Months' Exposure to Light

Oil no.	Window aspect	Odour	Spekker reading: Filter Violet 601 against water		Clarity
			Original	Exposed	
3	S	Very slight	<0·005	0·040	Very slight turbidity
8	S	Distinct	<0·005	0·040	Very slight turbidity
7	S	Distinct	<0·005	0·045	Slight turbidity
11	S	Strong	<0·005	0·050	Slight turbidity
1	S	Strong	<0·005	0·060	Slight turbidity
4	S	Strong	<0·005	0·086	Slight turbidity
5	S	Very pungent	<0·005	0·120	Slight white sludge
12 *	S	Very pungent	0·105	1·150	Orange-brown sludge
13 *	S	Very pungent	0·120	1·220	
2	W	Very slight	<0·005	0·012	Clear
6	W	Distinct	<0·005	0·077	Very slight turbidity
9	W	Distinct	<0·005	0·150	Very slight turbidity
10 *	W	Distinct	0·020	0·230	Slight turbidity

* Half-white oils.

The measurements of colour absorption were made on the Spekker absorptiometer in a 4-cm cell using water as standard. The readings are tabulated as for 1 cm length. The oils were filtered through filter-paper before examination.

Measurements of light absorption were made using the monochromatic filters Violet 601, Blue 602, Blue Green 603, and Yellow Green 605, corresponding with median wavelengths 4240 Å, 4650 Å, 4985 Å, and 5460 Å.

The most significant light absorption occurred with the violet filter, and these are the figures recorded in Table II.

In terms of visual colour the reading 0·020 corresponds with faint yellow, 0·120 with pale yellow, 0·230 with straw colour, and 1·150 and 1·220 with orange-brown.

Type of Examination Carried out on Exposed Samples

The introduction has indicated that the present investigation is concerned mainly with deterioration in quality as it affects the consumer. This involves inspection for changes in odour, colour, growth of visible globules, and separation of oil and water in emulsions.

The scope of the investigation might be extended, for example, to measuring the development of acidity, increase in peroxide value in the unhomogenized oils and, in emulsions, to the statistical treatment of globule sizes, and the measurement of the extinction coefficient of convenient dilutions in oil by the Spekker absorptiometer as carried out by Jellinek and Anson.[1] The measurement of viscosity changes in the creams might also be considered.

There are difficulties which render such extensive investigations undesirable. For example, the comparison of different oils and emulsions is best carried out without disturbance during exposure, and exposure should be of such duration that the quality as it appears to the consumer shows marked differences from one sample to another.

By the time such differences are manifest, most of the samples in the emulsified state show marked lack of homogeneity either in the form of oil-and-water separation or the appearance of very large globules. Any measurement of the properties of such a mixture, such as viscosity, would be without meaning.

The rate and extent of acid and peroxide development is likely to be influenced in these exposure tests by the fact that contact with air is restricted by closure of the container.

Some more extended examinations have, nevertheless, been made on emulsions which, after exposure, do not show pronounced visible heterogeneity.

Stability of Emulsions

The state of division of the disperse phase in an emulsion is an important factor influencing general stability of the emulsified system. Two water-in-oil emulsions were therefore made from each oil, one in which the globule size was relatively coarse, and one of a fine structure. The composition of each emulsion made was identical with reference to the percentage of oil, emulsifying wax, and aqueous phase,* and the conditions of preparation were as nearly as possible identical in each of the two sets.

The coarser structure was achieved by limiting the time of vigorous stirring to 5 minutes and the finer structure by extending stirring to 30 minutes and finally homogenizing. The differences in microscopic appearance are shown in Fig. 1.

* Oil : water phase = 1 : 1·33 V/V. Emulsifier wax : Oil = 6·75 : 100 W/V. Units—gms, mls.

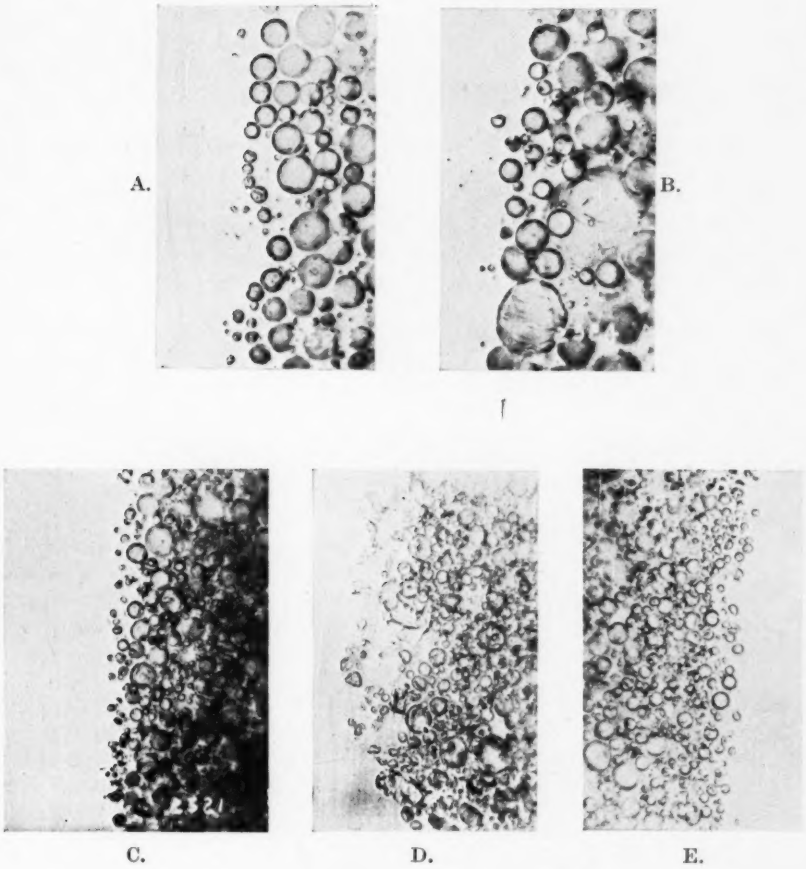
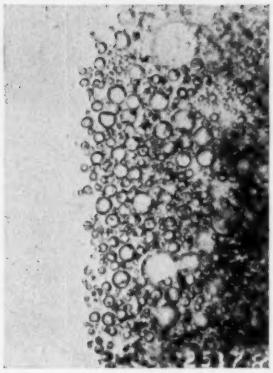


FIG. 1

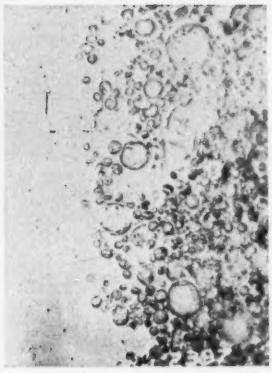
OIL NO. 2. (× 200)

A. Coarse emulsion stored in dark.
B. Coarse emulsion stored in light four months.
C. Fine emulsion stored in dark.
D. Fine emulsion stored in light four months, near surface.
E. Fine emulsion stored in light four months, below surface layer.

[*To face p.48.*



A.



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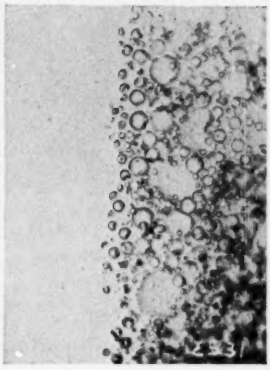

C.

FIG. 2

OIL NO. 6. (× 200)

A. Fine emulsion stored in dark.
B. Fine emulsion stored in light four months, near surface.
C. Fine emulsion stored in light four months, below surface layer.

The photomicrographs show that, under the influence of sunlight, a breakdown in the globular structure occurs at and near the surface of the cream, and that in the body of the cream a growth in globule size commonly occurs. The growth of water globules is progressive, and in time their ultimate coalescence leads to the formation of oil and water layers and a complete breakdown.

It is interesting to note that, when the emulsion develops a colour through exposure to light in the manner described, the yellowness appears first at the surface and spreads downwards as the layer in which globule breakdown occurs increases in depth. The thickness of this layer can be observed because the emulsion there becomes sometimes slightly more fluid than the main bulk. The growing layer sometimes quickly becomes a layer of clear oil, but frequently remains cloudy or opaque while it is warm, and it then normally sets on cooling with a sharp line of demarcation from the cream below it.

The development of colour from the surface downwards may be due either to the slow absorption of air standing above the cream or to the fact that the oil in the body of the cream is somewhat protected by the scattering of light from the globules, whereas near the surface the globules are being broken down and the light is not scattered to the same extent. The former hypothesis is supported by the fact that the pH of the creams decreases from the top downwards. In some cases the original pH of 10·5 to 11·0 drops at the top to 5 to 6, and at the bottom to 10 after four months' exposure.

Storage for one month in light in half-filled white glass jars gave striking differences between Nos. 5, 12, 13, and the rest, but there were no significant differences amongst the remainder, all of which developed a slight creamy coloration and an odour. No. 5 was pale yellow near the top, and 12 and 13 were yellowish brown.

Samples 2, 6, 9, and 10 definitely showed an increase in odour and a gradation in colour from white in 2, off white in 6, cream in 9, and 5 per cent deep yellow layer at the top of 10.

In the latter series, facing western light, the oils are more diverse in character than those in the first series, and differentiation after storage is thus a simpler matter.

The same differences were found in both the coarse and fine emulsions.

Effect of Four Months' Exposure of Emulsions to Light

The coarse emulsions placed in the window facing south had, with the exception of No. 3, broken down almost completely at the end of four months. No. 3 emulsion, although it had thinned considerably and was now pourable, showed no separation of water, and the globules were not visible to the naked eye. No. 8 had broken down to the extent that about 45 per cent of its volume contained large globules from 1 to 3 mm in diameter. The remainder of the emulsions showed similar breakdown to the extent of 55 to 65 per cent. In Nos. 12 and 13 the large globules had mainly collapsed to form clear, pale yellow water layers.

Colour deterioration was pronounced in Nos. 5, 12, and 13.

Fine Emulsions

A more definite grading was possible in the case of the fine emulsions, and after four months' exposure they fell into the order of progressive deterioration given in Table III.

The order of stability of the emulsified oils is not the same as for the unemulsified, in the middle of the series exposed in a south window. In the four oils in the west window the order is identical.

Stability of Emulsions in the Dark at 60° C

In view of the high temperature attained from time to time by samples exposed to direct sunlight, the coarse and fine emulsions were incubated at 60° C in the dark.

The coarse emulsions showed initially rapid separation of oil at the top, amounting in one day to approximately 5 per cent of the total emulsion, in five days to 14 per cent, and in seven days to 23 per cent. All the emulsions showed approximately the same oil separation.

TABLE III

Characteristics of the Fine Creams

Sample no.	Order of decreasing stability
	Stored facing South
3	White, semi-fluid, *ca* 3% visible globules at bottom, odour mild
8	White, fluid, *ca* 9% visible globules at bottom, odour distinct
1	Off white, fluid, 9% visible globules and 1% water
11	Off white, fluid, *ca* 12% visible globules and 1% water
4	Cream, fluid, full of large globules *ca* 0·5 mm dia top 2 mm at bottom
7	*ca* 10% water layer, 10% pale yellow oil
5	*ca* 15% water layer, 9% pale yellow oil
13	*ca* 20% yellow water layer, 9% straw-coloured oil
12	*ca* 40% yellow water layer, 20% straw-coloured oil
	Stored facing West
2	White, just pourable, no visible globules, odour mild
6	Off white, just pourable, no visible globules, odour strong
9	Cream, just pourable, no visible globules, odour strong
10	Cream, *ca* 14% large globules 2–3 mm dia at bottom, 14% yellow oil, odour pungent

There was progressive increase in the size of the globules at the bottom of the cream as shown in Table IV.

The fine emulsions were of better stability at 60° C than the coarse, showing from 13 to 17 per cent oil separation after seven weeks. In no case was there any separation of a water layer at the bottom.

The order of deterioration was not the same as for the coarse emulsions as shown in Table V.

After ten weeks' incubation the supernatant oil was poured off and measured. The remainder was centrifuged under standard conditions and the total oil and water separation tabulated in Table VA.

An examination of Tables II to VI shows that the oils and emulsions 2, 6, 9, and 10 decrease in light and heat stability as their aniline points decrease.

TABLE IV

Growth of Globules in Coarse Emulsions Incubated at 60° C for Seven Days

Oil no.	Size of globules at bottom, mm	Globules in cream
2	*ca* 0·5	Microscopic
8	*ca* 0·5–1·0	Microscopic
6	*ca* 1·0–1·5	Microscopic
9	*ca* 1·0–2·0	Microscopic
7	*ca* 1·0–2·0	*ca* 0·5
1	*ca* 1·0–2·0	*ca* 0·5 mm
3	*ca* 1·0–2·0	*ca* 0·5–1·0 mm
11	*ca* 2·0–3·0	—
10	60 per cent filled with 1 mm globules	
4	Water layer 20 per cent composed of large cells, *ca* 6 mm	
5	Water layer 25 per cent composed of large cells, *ca* 6 mm	
12	Water layer 25 per cent. No cellular structure	
13	Water layer 33 per cent. No cellular structure	

The emulsions all retained their original colour.

TABLE V

Fine Emulsions Incubated at 60° C for Seven Weeks

Emulsion no.	Separation of oil, per cent	Globular sizes
2	13	Few visible
3	13	Few visible
11	13	Few visible
6	15	All less than 1 mm and tabulated in order of increasing roughness of appearance of emulsion at glass wall
8	15	
1	17	
9	17	
7	17	
4	17	
5	17	
10	17	
13	17	A few water-pockets 5 mm dia
12	17	A few water-pockets 5 mm dia

TABLE Va

Emulsion Breakdown—10 Weeks at 60° C

Oil no.	% Water	% Oil	Total separation, %
2	0·7	12·4	13·1
11	3·7	12·6	16·3
3	6·1	14·2	20·3
8	8·2	14·7	22·9
7	1·3	23·1	24·4
6	1·7	22·9	24·6
4	8·2	17·1	25·3
9	5·8	22·5	28·3
10	2·9	26·1	29·0
1	9·2	20·4	29·6
5	4·4	28·2	32·6
12	4·0	31·8	35·8
13	4·0	31·8	35·8

TABLE VI

Peroxide Values of Oils Oxidized at 140° C for 1½ Hours and of Oils Exposed to Light for Four Months

Oil no.	Peroxide value × 10^4	
	Oxidized at 140° C	Exposed to light
11	5	96
12	13	27
13	14	20
2	15	54
3	17	66
1	37	70
4	68	43
8	86	—
5	145	15
7	183	—

In the nine other oils this simple relationship is complicated by the inclusion of oils of very different gravity and viscosity, and low specific gravity and viscosity in the oil allows freer movement of the aqueous globules in the emulsions and sedimentation of large globules. Close approach and coalescence of larger globules is thus facilitated. It is clear also that oxidation products formed on the surface in contact with air will diffuse more quickly into the body of the oil or emulsion, and the rate of general deterioration will be increased.

On the other hand, high specific gravity and viscosity will tend to stabilize the emulsion and also slow down the progress of oxidation both in the oil and in the emulsion.

Oil No. 1 has a much lower stability than would be expected from the aniline point, and this anomaly is attributable to the low specific gravity and viscosity as shown in Table I.

The gravity and viscosity of oil No. 8 are both above the average in Table I and the stability of the oil and its emulsions is higher than would be expected from the aniline point.

Laboratory Oxidation

The oils and emulsions invariably contain peroxides and free acid after exposure to light and air, and it was, therefore, considered desirable to carry out laboratory oxidations under defined conditions in order to seek some rapidly ascertainable characteristic of an oil which might serve to predict its behaviour under prolonged exposure to light and air.

The procedure adopted was to blow filtered compressed air through 50 ml of the oil in a glass tube immersed in a heating bath of glycerin. The air was metered at the rate of 4·8 litres per hour and was spread through the oil by passage through a sintered-glass disc immersed in it.

The determination of peroxide value was made by the method described by Yule and Wilson,[2] in which the oil under examination is shaken for 5 minutes with a solution of ferrous sulphate and ammonium thiocyanate in an acidified mixture of equal volumes of water and acetone. The ferric iron produced is titrated with N/100 titanous chloride solution. Solutions

were stored and dispensed in an inert atmosphere, and the titration flask was flooded with carbon dioxide. The peroxide value is calculated as mg equivalents of oxygen per gm of sample.

Preliminary experiments showed that oxidation was slow at temperatures in the region of 100° C and became conveniently rapid at 140° C. A standard time of 1½ hours was established by running oxidations for different times on two oils which were known to behave differently on

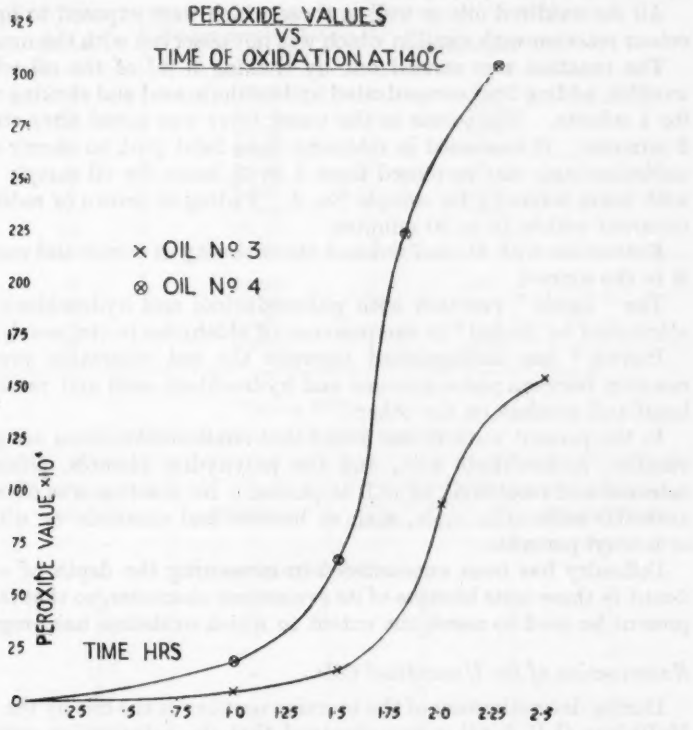

FIG. 3

exposure to light. The time adopted (1½ hours) was that at which the curve peroxide number *v* time had not yet reached its steepest slope. (See Fig. 3.)

The figures obtained for peroxide numbers do not place the oils or emulsions in the order of their observed stability to light, the notable anomalies being the half-white oils Nos. 12 and 13, which are placed on peroxide evaluation near the head of the stability table.

The measurement of acid values after oxidation again fails to afford a parallel classification.

Tests on the Oxidized and Exposed Oils

A single cold extraction of the oxidized oils with ethanol or methanol removed much of the colour and reduced the peroxide number to about

50 per cent of its value. The alcohol extract was found to contain approximately the balance of peroxide and colouring matter.

Kreulen and Kreulen van Selms [3] have reported that a fair indication of the degree of oxidation of a mineral oil can be obtained by shaking it with 96 per cent alcohol and recovering the dissolved fraction. There is evidence that, in addition to extracting substantially the peroxides and the hydroxy acids postulated by these authors, the alcohol also removes something in the nature of a polyhydric phenol.

All the oxidized oils as well as those which were exposed to light gave a colour reaction with vanillin which was not observed with the untreated oil.

The reaction was carried out by shaking 5 ml of the oil with 10 mg vanillin, adding 3 ml concentrated hydrochloric acid and shaking vigorously for 1 minute. The colour in the water layer was noted after standing for 2 minutes. It increased in intensity from faint pink to cherry red as the oxidation time was increased from 1 to 2½ hours for oil sample No. 4 and with lower intensity for sample No. 3. Fading to brown or reddish brown occurred within 15 to 30 minutes.

Extraction with alcohol reduced the intensity of colour and concentrated it in the extract.

The " lignin " reaction with phloroglucinol and hydrochloric acid was attributed by Nickel [4] to the presence of aldehydes in the wood.

Powick [5] has distinguished between the red coloration produced by reaction between phloroglucinol and hydrochloric acid and vanillin on one hand and acrolein on the other.

In the present work it was found that similar colorations are given with vanillin, hydrochloric acid, and the polyhydric phenols, phloroglucinol, catechol and resorcinol, as well as phenol. No reaction was obtained with aromatic carboxylic acids, such as benzoic and cinnamic or with benzoyl or lauroyl peroxide.

Difficulty has been encountered in measuring the depth of colour produced in these tests because of its evanescent character, so that it cannot at present be used to assess the extent to which oxidation has progressed.

Examination of the Unoxidized Oils

During determination of the bromine number of the oils by the method of McIlhiney (I.P. 9/42) it was observed that the substitution occurring was low for the oils of highest light-stability and higher for low-stability oils.

As a method of grading this would be open to the objection that it is difficult to obtain either reproducibility or repeatability. Nevertheless, bromine substitution numbers were calculated from duplicate or triplicate determinations after eliminating a few values here and there associated with negative bromine numbers.

The bromine substitution number is calculated from the amount of hydrobromic acid liberated during the estimation of bromine number by method I.P. 9/42, in other words it is derived from the correction factor to be applied in the calculation of bromine number.

Aniline Points as a Means of Classification

The determination of aniline points by the method A of I.P. 2/45 affords a simpler and more reliable means of classifying the white and half-white oils

with regard to light stability. The results given by the bromine substitution numbers and the aniline-point method are compared with the order of deterioration of the oils and emulsions after four months' exposure to light in the Tables VII and VIII.

TABLE VII

Bromine Substitution Numbers and Aniline Points

Order as in Table II

Oil no.	Bromine substitution no. g Br per 100 g oil	Aniline pt, ° C
3	0·017	99·2
8	0·013	95·8
7	0·024	92·8
11	0·023	95·9
1	0·008	97·4
4	0·020	95·6
5	0·046	91·8
12	0·178	80·0
13	0·167	79·8
2	0·008	99·7
6	0·028	95·7
9	0·046	91·8
10	0·075	90·0

TABLE VIII

Order of Decreasing Light Stability

Oil	Coarse emulsion	Fine emulsion	Order of increasing bromine substitution no.	Order of decreasing aniline pt
		Exposed to South Light		
3	3	3	1	3
8	8	8	8	1
7	7	1	3	11
11	11	11	4	8
1	1	4	11	4
4	4	7	7	7
5	5	5	5	5
12	12	13	13	12
13	13	12	12	13
		Exposed to West Light		
2	2	2	2	2
6	6	6	6	6
9	9	9	9	9
10	10	10	10	10

Fluorescence

The half-white oils under examination had a pronounced bloom in daylight, and their stability to light was of a low order. The white oils exhibited no fluorescence in daylight, but under the ultra-violet lamp all fluoresced to some extent. The oils were graded according to the intensity of fluorescence, and a close relationship is shown between this classification and the order of light stability.

TABLE IX

Fluorescence under Ultra-violet Lamp

Oil no.	Fluorescence	Oil no.	Fluorescence
2	Very faint mauve	4	Strong bluish
3	Faint greenish	9	Strong bluish
1	Faint greenish	5	Strong bluish
7	Moderate greenish	10	Very strong bluish
11	Distinct greenish	12	Intense bluish
6	Distinct bluish	13	Intense bluish
8	Distinct bluish		

The emulsions exhibit a similar fluorescence.

Ring Analysis

An approximate ring analysis of some of the oils under investigation has been carried out by making measurements of the specific refraction of Lorenz–Lorenz $\frac{n^2-1}{n^2+2}\frac{1}{d}$, where n is the refractive index at 20° C for sodium light and d the specific gravity at 20° C against water at 4° C.

In conjunction with molecular weights measured cryoscopically in *cyclo*hexane and aniline points, the graph relating molecular weight with specific refraction and aniline point for aromatic-free oils given by Vlugter, Waterman, and van Westen [6] was used to calculate the rise of aniline point to be expected as a result of complete hydrogenation instead of determining this by actual hydrogenation. The composition of the oil was expressed in the manner suggested by these authors in terms of percentage aromatic rings, naphthenic rings, and paraffin side chains.

TABLE X

Approximate Ring Analysis

Oil no.	Specific refraction, $\frac{n^2-1}{n^2-2}\cdot\frac{1}{d}$	Mean mol. wt.	Aromatic, %	Naphthenic, %	Paraffinic, %
2	0·3263	340	1	29	70
3	0·3262	345	2	29	69
8	0·3238	345	2	37	61
9	0·3238	320	2	40	58
11	0·3228	370	2	41	57
4	0·3254	340	3·5	31	65·5
7	0·3238	335	3·5	35·5	61
5	0·3246	330	4·5	34	61·5
10	0·3238	330	5	39	56

EVIDENCE OF THE INFLUENCE OF AROMATICS ON STABILITY

The stable oils Nos. 2 and 3 are characterized by low aromatic content and a high paraffin/naphthene ratio, whilst the unstable oils, 5 and 10, show high aromatic and a lower ratio.

Although the bromine-substitution values are not measurable with precision, there is a general drift in the values obtained under carefully con-

trolled conditions from low values for the most stable oils to high values for the unstable ones. Bromine substitution may indicate aromatic rings or branched paraffinic chains with tertiary carbon atoms or naphthenes with paraffinic chains and possessing, therefore, tertiary carbon atoms.

The ultra-violet-absorption spectrum of oil No. 9 (Fig. 4) showed a maximum absorption at mμ 271 and exposure of the oil to light and air for four months caused a shift in the maximum to mμ 265. This also may indicate that aromatic components in the oil undergo change during exposure to light and air.

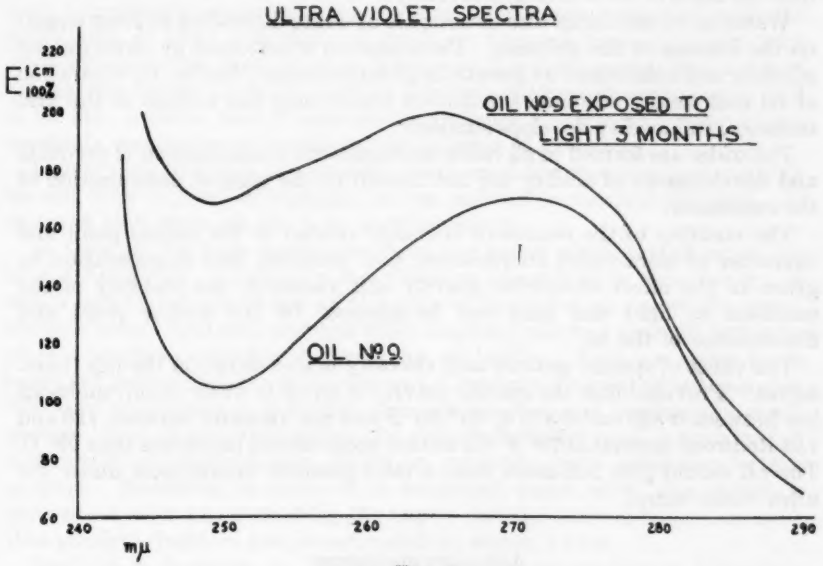

FIG. 4

A further indication that aromatic components may be responsible for instability is provided by examination of the fluorescence of the original oils under the ultra-violet lamp (Table IX).

Opinion is divided as to the effect of aromatics on the accelerated oxidation of oils at temperatures above 100° C. Larsen, Thorpe, and Armfield [7] state that although simple aromatics are very reactive, polynuclear aromatics and naphthenes are very stable.

Wood-Mallock [8] inclines to the view that sulphur compounds present in the oil act as oxidation inhibitors, and when these are removed the oil is unstable even when the aromatic content has been reduced to low percentage.

On the other hand, Kapff, Bowman, and Lowry [9] consider that susceptibility to oxidation (acid development) of close cuts under the conditions of experiment is apparently dependent upon the chemical constitution as indicated by the Waterman analysis and is not to be imputed to the presence or absence of natural antioxidants.

Conclusions

White oils exposed to light in contact with air deteriorate in odour, colour, and clarity to an extent which appears to be closely related to the aniline point of the oil and the nature of the fluorescence produced by ultra-violet light. Peroxides and free acids are developed during the deterioration, but the stage of development is not a measure of the general deterioration. A low specific gravity and a low viscosity both tend to cause more rapid deterioration, presumably because migration from the surface into the mass of oil occurs more readily.

Water-in-oil emulsions also deteriorate at a rate depending to some extent on the fineness of the globules. Deterioration is indicated by development of odour and colour and by growth in globule size and, finally, by separation of oil and water. Globule breakdown occurs near the surface in the first instance, and acidity develops similarly.

Peroxides are formed in all cases, and again the concentration of peroxide and development of acidity are not related to the general deterioration of the emulsions.

The stability of the emulsions is closely related to the aniline point and character of ultra-violet fluorescence, and, provided that consideration be given to the effect of specific gravity and viscosity, the stability of the emulsion to light and heat can be assessed by the aniline point and fluorescence of the oil.

The effect of specific gravity and viscosity is also shown in the oils themselves. Provided that the specific gravity of an oil in water-in-oil emulsions lies between 0·885 and 0·875 at 60°/60° F and the viscosity between 120 and 155 Redwood seconds at 70° F, the aniline point should be not less than 98° C. The oil should give not more than a faint greenish fluorescence under the ultra-violet lamp.

Acknowledgments

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Discussion

The Chairman: I am sure we want to thank Dr Myddleton for his presentation of his paper, and I hope we shall have an interesting discussion on it. Amongst us here to-night we have Mr Biske, who is a Member of the Council of the Institute, and I am going to ask him if he will open the discussion.

V. Biske: The subject which Dr Myddleton has dealt with this evening, namely the stability of white and half-white mineral oils, is one which is of very considerable importance to those of us who are connected with the production of these materials. I think we have all listened to him with close attention, the more so since I am sure it will be agreed that some of his statements are probably open to a certain amount of controversy, and I, for one, consider that if some of the audience disagree with some of the speaker's conclusions it heightens the interest of a meeting.

I note that although the speaker has named the months during which his oils were exposed to sunlight, he has omitted to mention the year. If this was 1950, then his oils were let off lightly!

In view of the large deviations from the mean which are observed, at least subjectively, in intensity and duration of sunlight in this country, it would be advisable for a test of this type to be placed on a partially quantitative basis. Our own practice when exposing oils to a sunlight test is to place a sunshine recorder alongside the oil bottles. Such recorders do at least give some indication of the amount of sunlight received by the samples. The type we employ is a Jordan, this is a simpler, and incidentally much cheaper, type of instrument than the Campbell-Stokes used as standard in meteorological observatories, but it is probably quite adequate for the purpose. Recording is done on a sensitized paper strip, the record is permanent after water-washing the paper, and the scale divisions are such that sunlight duration can be estimated to within $\frac{1}{4}$ hour.

Sunlight is, however, in Britain at least, an unsatisfactory laboratory reagent. We have found that a useful indication of the stability of a white oil can be obtained by exposure for two hours at a short distance from an ultra-violet lamp; such exposure is, of course, done in a quartz flask. After such time most oils show a measurable increase in colour as determined by the W.O.M.A. number, the extent of increase being an indication of stability. It is not clear whether the mechanism of such colour change is the same as that causing colour deterioration in sunlight. It would hardly seem likely that the latter can be due to ultra-violet light, since, in the case of a bottle exposed in a shop window, the sunlight is filtered through two thicknesses of ordinary glass, which is opaque to ultra-violet radiations.

As regards fluorescence, we have been hoping to try the procedure described by Kinsel and Phillips * for the qualitative assessment of the type of fluorescence, but have not so far had an opportunity of constructing the fairly simple instrument described. It would be interesting to know if either Dr Myddleton or any one else here has any experience of this test; the procedure given by the authors is for visible light, but should presumably be easily adapted to ultra-violet illumination.

* *Refiner*, 1950, **29** (5), 93.

I note that Dr Myddleton is not in favour of peroxide value as an indication of photo-stability. I should like to ask him whether he has any opinions to express on the work of Golden,* who claims that a qualitative detection of the presence of peroxides after heating the oil to 300° F can be used to predict "shelf-life," the criterion used by this author being the time for which the oil may be heated at this temperature before peroxides are found. According to his results, if the oil withstands heating for 10 minutes at this temperature (*i.e.*, shows no peroxides after this period), this is equivalent to the satisfactory shelf-life of ten months. So far our own tests on correlating peroxide number determined by this method with stability to sunlight have not been carried on long enough to express a definite view on this point.

On this question of shelf-life, it may be of interest to mention that the latest (XIVth) edition of the *U.S. Pharmacopoeia* permits addition to liquid paraffin of up to 10 p.p.m. of tocopherol as a stabilizer.

Finally, I must confess to being a little puzzled by the speaker's results on bromine numbers. The degree of accuracy which he reports is far greater than that which can be expected of I.P. method 9/42 which he uses; and which gives a repeatability, at the best, of 0·1. I notice that Dr Myddleton refers to bromine *substitution* number; I think that bromine number is usually considered to be a measure of bromine *addition*. Perhaps he could explain this point.

Whilst I have been very interested to study Dr Myddleton's results, I must admit that I am not fully convinced by his arguments. For the time being at least, I would prefer to assess white-oil stability by a more direct method, such as, for instance, darkening after exposure to ultra-violet light, or, in the case of medicinal grades, by comparing the results of the B.P. hot-acid test on an oil before and after it has been heated to 100° C for 16 hours with access of air, which well-known procedure (see Archibald, "Science of Petroleum," 1938, IV, 2839) appears to give quite a reliable indication, rather than by determination of bromine number or aniline point.

However, I should like to express my thanks to Dr Myddleton for his very stimulating paper; it is seldom that we have an opportunity of discussing this subject at an Institute of Petroleum meeting, at least from the very practical angle from which to-night's speaker has approached it, and I feel sure that the views expressed in the discussion now starting will do much towards clarifying this still somewhat empirical subject.

DR MYDDLETON : I am very much obliged to Mr Biske for his helpful remarks and criticisms. To take his first point with regard to the period of the tests : they were carried out during 1950 from April onwards, but I regard this point as of little importance to the general issue. All the oils and emulsions exposed to south light were subject to the same conditions and so also were those exposed to west light, and so long as we do not compare the effects produced in one group with those produced in the other we can dispense with the elaboration of providing equipment to measure the duration and intensity of the light which produced the effects. If these measurements had been made the results would be no nearer quantitative

* *J. Amer. Pharm. Ass.*, 1946, **35**, 76.

in the sense that other observers could repeat the precise effects by exposing the oils to light of the stated intensity and duration. Before this could be done some information must also be forthcoming as to the nature of the glass of the window and container, and also as to the temperature variations during the exposure.

It may quite well be that the mechanism of deterioration under the influence of ultra-violet light is not the same as in sunlight. That alone would not invalidate the use of ultra-violet light for the rapid assessment of sunlight stability provided that some correlation of effects could be shown. The fact is, however, that when the oils used in these tests were exposed in shallow dishes without covering, for 60 hours, 6 inches below an ultra-violet lamp, no change in colour measurable by the Spekker absorptiometer was observed, except in the case of the half-white oils.

The qualitative assessment of the type of fluorescence is certainly desirable, and although it was planned to make use of the Spekker fluorimeter for the purpose, I hope to explore the possibilities of the method to which Mr Biske has drawn attention.

The peroxide values I have quoted were measured on oils which had been oxidized at 140° C, some 9° lower than the temperature used by Golden. At the lower temperature the induction period is somewhat longer than at the higher temperature, and the measurement of peroxide value after 1½ hours oxidation in our procedure is not substantially different from measurement after 10 to 15 minutes as in Golden's procedure. Amongst the white oils we have examined, some are undoubtedly placed in correct order of decreasing light stability when arranged in order of increasing peroxide value, but a significant number are out of order. There is the further complication that the peroxide value of an oil which has been exposed to sunlight is no measure of the general deterioration which the oil has suffered in terms, for example, of colour and odour. Similarly, peroxide value is no measure of the deterioration which has taken place in an emulsion in terms of colour, odour, growth and coalescence of globules, and water and oil separation.

The use of stabilizers to protect oils from oxidation and other forms of deterioration is a matter of considerable importance, and, although α tocopherol may be one of the most efficient, it is, in my opinion, of paramount importance to make sure that the oil selected is characterized by high stability.

A stabilizer is at the best a temporary remedy to extend the induction period before the onset of pronounced deterioration.

Mr Biske asks for a clarification of the term "bromine substitution number." The term is what its name implies, namely the number of gm of bromine combining with 100 gm of oil in substitution reactions as distinct from addition. It is derived from the measured amount of hydrobromic acid liberated, for example, in the I.P. method 9/42. The substitution effect is influenced by sunlight and by variation in temperature, and concordant results can be obtained only under identical lighting and temperature conditions. For this reason the determination of bromine substitution numbers is not recommended as a normal test for stability.

The B.P. hot acid test is open to some objection because it is impossible

to ensure standard conditions of contact between the acid and oil. Hand shaking is difficult to standardize and, moreover, the two phases separate at different rates in different oils in the intervals between shaking. In the B.P. method the intervals are of 2 minutes' duration, and in this feature the method described by Archibald is an improvement with ½-minute intervals.

It is to be noted that Archibald requires the test to be carried out after the oil has been oxidized for 16 hours, and this introduces a delay which is frequently inconvenient. The aniline point can be determined accurately within 90 minutes, including 60 minutes for the drying of the sample.

There would still be a strong argument in favour of the hot acid test on the oil after heating if it were capable of placing the light oil No. 1 in correct position amongst the other oils with regard to light stability. This test, however, places No. 1 between Nos. 2 and 3 as the most stable three amongst those examined. The order of decreasing stability given by the test is 2, 1, 3, 11, 8, 7, 6, 5, 4, 9. The half-white oils were not submitted to the test.

DR R. SCHNURMANN : I am a little puzzled by Dr Myddleton's proposal to introduce the aniline point as the stability specification for liquid paraffin and for technical white oils. If I followed Dr Myddleton correctly, the position is that he examined certain oils, and he showed us on one slide nine of these thirteen oils in a definite order. Now I believe that the position is this, that there are two *practical* criteria of whether the oil is stable or not, and one is, as Dr Myddleton said, the colour, and the other one is the odour (one might perhaps add the taste) of the oil, and I fail to see how, from these criteria, one can put nine oils into a definite order. Quite apart from the fact that there is the complicating factor of having then to consider variations in specific gravity and viscosity, and I also think that among these thirteen oils examined by Dr Myddleton there is only one of the ordinary liquid paraffins—at least if one goes by his viscosity data.

Now we know perfectly well in the oil industry that we can have liquid paraffins which all have practically the same viscosity and the same specific gravity and still some of them are extraordinarily stable and some of them are very unstable, to the extent that after a short exposure to sunlight they develop an odour, or they may even develop a discoloration.

Dr Myddleton showed in one of his slides the ultra-violet-absorption spectrum of a technical white oil, and we believe at the Manchester Oil Refinery that this is one of the possibilities of laying down, at the moment, a specification for the stability of a white oil. The position is this, that every liquid paraffin and most of the technical white oils exhibit a very similar ultra-violet spectrum to the one shown by Dr Myddleton for his oil number 9 : that is a plot of the absorption intensity against wavelength with a minimum, if it is a stable oil, in the neighbourhood of 2490 Å, with a maximum in the neighbourhood of 2710 Å, and with a subsidiary maximum at about 2780 Å, and from a great deal of material that we have collected it seems quite clear that if the ratio of the absorption intensities at this maximum 2710 Å and at the minimum of 2490 Å is above a certain figure and if the absorption intensity at 2710 Å does not exceed a definite value, then one can quite clearly say, " This is a stable oil and the others are not."

DR MYDDLETON : I would like to thank Dr Schnurmann also for his very helpful contribution to the discussion. The grading of a number of oils by odour is admittedly difficult, and it will be noted that the nine oils which were exposed to south light are divided into four odour categories and also four with regard to clarity.

With regard to colour, these oils were graded by eye in the first instance and were then examined by the Spekker absorptiometer by an independent observer. The results were identical.

The stability of emulsions is perhaps more important to the toilet and cosmetic industry than that of the oils alone, and I think I have given some evidence of a close relationship between the one and the other, so that a study of oil stability must be extended to cover emulsions also. In the emulsions, of course, other kinds of deterioration are measurable.

Dr Schnurmann considers the factor of specific gravity and viscosity to be an objection to the adoption of aniline point as a criterion of stability. I have pointed out that the toilet and cosmetic chemist is limited to certain narrow ranges of gravity and viscosity to achieve certain objects in using the oil. He is then able to use aniline points without further complication.

With regard to liquid paraffins, I would venture to suggest that aniline points would again serve to distinguish the stable from the unstable oils in this class.

R. M. FREMOUTH : I should like to ask Dr Myddleton if all the experiments with the emulsions were conducted in an emulsion where the oil was the continuous phase, or did he conduct collaterally a series of tests where the water was the continuous phase, and does he think that the aniline point would affect the latter ?

DR MYDDLETON : The water-in-oil emulsions were studied in detail because it was considered that the action of light on the oil phase would be more pronounced than in the oil-in-water type, where much light is scattered from the surfaces of the globules.

If this be the case, then the deterioration due to oil instability will not be so rapid in the oil-in-water type, although oxidation effects will ultimately occur through the action of oxygen dissolving in the aqueous phase. The aniline point must still be regarded as a criterion of the stability of the oil.

DR A. S. FREEBORN : Dr Myddleton has spoken of water-in-oil emulsions, and the anti-oxidants which he has used are presumably dissolved in the oil. With oil-in-water emulsions would he expect a more beneficial effect with an anti-oxidant added to the water ? I might naïvely suggest sodium sulphite. Would he expect that to have a beneficial effect or not ?

DR MYDDLETON : The addition of an anti-oxidant to the aqueous phase of an oil-in-water emulsion would undoubtedly delay deterioration of the oil phase by countering the action of dissolved oxygen in the aqueous phase.

Whether the sulphite would prove objectionable in the application of the emulsion for the purpose which it is intended to serve is another matter.

F. GROSSMITH : I appreciate that Dr Myddleton's work is related to the colour stability, odour stability, and emulsion stability of mineral oils, and his conclusions have led him to a certain type of specification. I wonder if he has considered another important point, as to whether his type of specification also fits in with the Twort specification concerning the carcinogenic properties of these oils which, of course, for toilet preparations is a very important feature. It would be a pity if one specification conflicted with the other.

DR MYDDLETON : In reply to Mr Grossmith I think that the limit of the Twort specification is not approached at all by the white oils which we are considering. I have not yet found an example of anything approaching the danger line of the Twort specification with regard to carcinogenic properties in white oils, but there is this point to be made, that the oils which have already become oxidized and have deteriorated may come within the category of the dangerous oils according to the Twort method of examination, but provided that you get an original oil which does not go through that objectionable stage of deterioration you are doubly safeguarded.

H. W. STEVENSON : I have been connected with recent work on formulating a new specification for non-carcinogenic mule-spindle oils, and in actual fact the Twort specification has been discarded. It will be replaced by a new specification drawn up by a Manchester Committee in which the I.P. Research Group No. 2 have participated. I do not think, therefore, that the point raised by the speaker really applies nowadays. The new specification calls for a highly refined oil, and such an oil can be expected to have a high aniline point. The new specification is much more specific for non-carcinogenic mule-spindle oils than was the Twort specification.

R. A. FRASER : There are one or two points which I should like to ask Dr Myddleton. First, he has suggested that the specification for an oil for this toilet and cosmetic work should be based on certain properties of the oil. I believe one of the problems with which the cosmetic chemists have to deal is the incompatibility of essential oils with mineral oils, and I would like to know if his suggested specification covers that point as well.

Secondly, as the main criterion appears to be the stability of the emulsions formed by the oil, would it not be of interest to determine the interfacial tension of the oxidized oil against water?

DR MYDDLETON : It is a well-known fact, of course, that essential oils which require to be dissolved in mineral oils do need very special selection, and it is very difficult to guarantee that a given perfume made up of a number of different essential oils is going to be satisfactory. As far as the viscosity and gravity ranges that we have explored would be concerned, the aniline points suggested—the high aniline points—would not lead to any greater incompatibility with the essential oils.

I think it certainly would be useful to explore the interfacial tension of the oxidized oils against a water surface, for example, and it would lead to further information—perhaps quite valuable—on the stability of emulsions. It might even lead to the discovery of some way of counteracting any

variation and any drift in the interfacial tension that may be found to occur during oxidation.

E. S. RAWSON : I would like to ask Dr Myddleton if he would expect the aniline number to be a useful index of stability of emulsion made with, say, the same set of oils but with a different emulsifying wax. Perhaps I might ask the significance of the type of wax that is used ?

DR MYDDLETON : I pointed out that the emulsifying wax used in these experiments is slightly anti-oxidant, and there are other emulsifying waxes with about the same anti-oxidant properties and there are some with just none at all. They may even tend to accelerate oxidation, so I would think that by using the wax which has a slight anti-oxidant action, as we have in fact done, we would have a very reliable guide as to the serviceability of the aniline point in determining the stability of the oil.

W. POHL : I am not quite clear, in the course of the discussion, what exactly we are talking about in relation to the stability of the emulsions. From the paper I gathered that the subject was the stability of the emulsion to light in respect of the effect of light on the oil, but the discussion seems to have wandered on to the stability of the emulsion from the point of view of phase separation and globular size. Is the author claiming that the aniline-point specification bears relation only to the light stability of the oil in the emulsion, or does he also claim some relation to the phase stability and the globular size of the emulsion, or have we all got mixed up in the discussion ?

DR MYDDLETON : The effect of light on the emulsion is to break down the phases eventually, but the separation of an emulsion at room temperature is not affected, I think, by the aniline point of the oil used in its composition except perhaps on very prolonged storage. The aniline point, as far as I can see, will relate to the stability of the emulsion in sunlight or in heat in contact with a certain amount of air, and to very prolonged storage at room temperature. If you expose the emulsion to light and heat for a sufficient time, then you do get this phase breakdown, but I do not think it is of the same mechanism or significance as a breakdown which occurs when an emulsion is badly made and the interface is not properly formed and it is then allowed to stand at room temperature. That kind of a breakdown is something quite different.

O. H. PEEL : There is one small question which I should like to ask, and that is whether any sort of standardization level is possible by working on a synthetic paraffin with the maximum possible aniline as a basis for reference.

DR MYDDLETON : Yes, I am sure quite a lot of very valuable work could be done in that direction by using specified pure hydrocarbons, but we have not got the resources of the petroleum technologists. If we could get this type of product for examination we should be very glad to do so, and I think it would be very useful.

THE CHAIRMAN : If there is no further discussion I should like, on your behalf, to thank Dr Myddleton for his paper this evening. I have been very interested in this discussion, and I call upon you to accord a hearty vote of thanks to Dr Myddleton for coming here to-night and giving us this paper, and I will ask you to record that in the usual way.

(The vote of thanks was carried by acclamation.)

DR MYDDLETON : I thank you very much for your kind reception of this paper to-night, and I do appreciate the lively interest that was taken in the discussion, the very useful comments that were made, and the necessary criticisms that were passed. It has been a great pleasure to me to be able to come to the petroleum technologists and indicate to them some of the problems which we are facing, and we hope that the end of some of our difficulties will be in sight through your kind co-operation. I thank you all very much for your kindness.

DIFFUSION AS A RATE-LIMITING FACTOR IN THE OXIDATION OF TRANSFORMER OILS

By R. Irving and C. N. Thompson (Associate Fellow)

Summary

The rate of access of oxygen to oil in transformers is limited by constructional (and other) features, and the rate of oxidation of the oil probably depends primarily upon this factor. Under such conditions, oils with oxidation rates above a certain limit will oxidize at the same rate and not at the different individual rates which would occur in the presence of excess oxygen. The relative rates of sludge and acid formation in various transformer oils and related products have been determined under conditions where the rate of oxygen diffusion limits the rate of oxidation. The reaction rates and product proportions were significantly different from those observed in accelerated laboratory tests under similar conditions but with excess oxygen. In particular, slower rates of oxidation were observed to result from limited oxygen access, and a slow sludge-formation process became evident. The catalytic effects of soluble copper and iron on the oxidation rates of various types of transformer oil have also been investigated.

Introduction

From considerations of the conditions under which transformer oils oxidize in service it appears probable that the rates of oxygen absorption (and therefore oxidation) are controlled largely, or perhaps even entirely, by the ease of oxygen access to the oil. This, in turn, is governed by the effects of numerous constructional and other features, *e.g.*, breathing facilities presence or absence of a conservator tank, temperature gradients within the transformer, and oil circulation by convection currents. Evidence in support of this view has been discussed by Hurworth,[1] and is strengthened by consideration of the effective life of a transformer oil in service (fifteen years and upwards).[1,2] This time is so long compared with laboratory oxidation tests (even at temperatures approaching the operating temperatures of transformers) that the oxidation reactions in service must be governed very largely by the ease of oxygen access to the oil.

It is therefore possible that oils which have absolute oxidation rates * above a certain limit (this limit being imposed by the rate at which oxygen is made available to the oils) will absorb oxygen at identical rates if compared in identical transformers. That this does not appear to occur in service is due to: (*a*) the multiplicity of transformer constructions and operating conditions, and (*b*) the fact that oil oxidation in transformers is invariably assessed by the amounts of sludge and acidity. These may be either end-products of a series of oxidation and/or polymerization reactions, or may result from oil contamination by transformer constructional materials.

In order to acquire knowledge of the effects of a limited availability of

* That is, under oxygen excess conditions.

oxygen on the oxidation rate, various transformer oils and related products have been oxidized under conditions which may simulate, to a certain extent, those obtaining in a transformer in operation; these conditions are such that the rate of oxidation is controlled by the rate of diffusion of oxygen into the oil. The course of the reaction has been followed by measuring the *rate* of sludge and acidity development rather than the *extent* after a fixed time as in the B.S. 148 : 1933 sludge test. A more informative comparison between different oils is then possible.

Experimental

A number of samples (usually six) of each oil was placed into test-tubes whose dimensions (length, 6 inches, internal diameter, $\frac{3}{4}$ inch) were standardized in order to minimize errors due to varying surface area, oil depth, etc.* The required weight of oil was dispensed into each of the tubes, using a hypodermic syringe; after calibration once for each oil, this was found to deliver the correct amount of oil rapidly.

The test-tubes containing the oils under examination were supported in a specially constructed rack (dimensions 11 × 11 × 5 inches) which was capable of holding a maximum of sixty-four test-tubes in eight rows of eight tubes. The rack was then placed in an electrically-heated oven (internal dimensions 2 feet × 1 foot 4 inches × 1 foot 2 inches) the temperature of which was thermostatically controlled at 120° ± 1° C. Temperature gradients inside the oven were reduced as much as possible by fitting a three-bladed air-circulating fan centrally through the roof of the oven. The fan (5 inches radius) rotated at *ca* 500 r.p.m. about 1 foot 3 inches above the surfaces of the oils, which, in turn, were about 8 inches from the base of the oven. Although air-vents as such were not fitted in the walls of the oven, ventilation occurred through the space surrounding the shaft from the fan to an electric motor mounted on the top of the oven.

A temperature of 120° C was selected, as it is generally felt that 150° C (as used in the British transformer-oil oxidation test) is too high, and it has been found by other workers that a temperature of 120° C (or lower) is more suitable for research purposes.[3,4,5] Samples were periodically removed and examined for acidity formation and sludge development.

Sludge values were obtained by diluting each sample of oxidized oil with 2 volumes of I.P. petroleum spirit (60°–80° C) to one volume of oil,† allowing the mixture to stand in the dark at room temperature for 24 hours, and then removing the insolubles from the oil/petroleum spirit solution by centrifuging. After washing by decantation and centrifuging, the sludge was dried at 120° C for several hours and weighed. Acidities were then determined volumetrically, using an alcoholic solution of Alkali Blue 6B as a diluent, on the combined sludge-free oil–petroleum spirit solution and washings.

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* With one exception the densities of the oils studied range from 0·7967 to 0·8119 at 120° C. This range corresponds to a maximum difference in oil depth of *ca* 0·07 cm for a sample weight of 10 g. If the remaining oil (F, an unrefined spindle distillate) of density 0·8510 at 120° C is included, the maximum difference in oil depth is 0·25 cm for the same sample weight.

† This ratio was chosen so that the total volume of oil/petroleum spirit solution did not exceed the volume of the test-tube in which the oxidation was carried out.

This procedure obviates the complications arising from sampling from a single reactant system. The latter procedure is undesirable, because: (*a*) the samples withdrawn may not be homogeneous, and (*b*) each successive sampling will alter the ratio of oil volume to area of oil exposed to oxygen. The reaction equilibrium may therefore be seriously disturbed, and anomalous results may well be expected.

Experiments were carried out in the presence and absence of catalysts. It is difficult to prepare a solid metallic catalyst (*e.g.*, copper) having a fixed surface area which is at the same time regular and reproducible. It has been stated by Pollitt [6] that, after oxidation has commenced, other factors appear which influence the rate of oil deterioration, such as metal salts of organic acids. It was found in earlier work in these laboratories that transformer oils, after oxidation under conditions of the B.S. 148 : 1933 sludge test (standard conditions: reaction time 45 hours, temperature 150° C, solid copper foil catalyst, air-flow rate 2 litres/hour), contained significant amounts of chemically combined copper (of the order of 50 parts per million of oil) in the form of salts and organic complexes. Such amounts of copper in an oil-soluble form have powerful catalytic effects, and are possibly sufficient to determine the oxidation rate. Soluble metallic catalysts have been used by other workers.[7,8,9] In the present work, therefore, attention has been primarily directed towards the study of oil-oxidation processes in the presence of soluble catalysts. Whilst most of the work involved the use of copper (as naphthenate) * as the catalytic agent, some investigations were made using the corresponding iron compound and mixed copper/iron catalysts.

Results

As a necessary preliminary to this investigation, the effect was studied of varying the oil-sample weight on the relative rates of sludge and acidity development, using the technique described above. Fig. 1 summarizes graphically the results which were obtained for two transformer oils in the presence (oils C and D) and in the absence (oil D) of a soluble copper catalyst.

In all cases the effect of increasing the sample weight from 5 to 20 g was to decrease the rate of deterioration thus affording strong evidence in favour of the view that diffusion is a rate-limiting factor under conditions where the geometry of the system is such that the ratio of oil depth to surface area is large. Experiments involving the oxidation of thin films of oil of relatively large surface area showed that the rate of deterioration (as measured by the rate of oxygen absorption) was not, under these conditions, predominantly controlled by a diffusion process over a range of oil-film depths. However, as the amount of oil was increased, diffusion became operative as a rate-determining factor, this being evidenced by a slower oxidation-reaction rate.

* The copper naphthenate used was prepared by a double-decomposition reaction, in the presence of low-boiling-point petroleum spirit, between copper sulphate and sodium naphthenate. The latter salt was prepared using a carefully de-oiled and distilled fraction of naphthenic acids. Samples of copper naphthenate having copper contents of the order of 6 to 12 per cent weight have been shown to have identical catalytic effects, providing the catalyst addition is made on a copper-weight basis.

It therefore appears that under the " test-tube " oxidation conditions, deterioration takes place in the " diffusion-controlled " region; a fairly

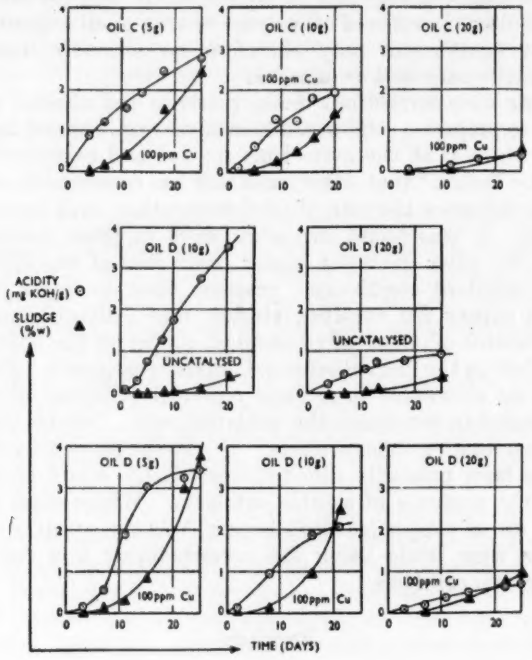

FIG. 1

EFFECT OF SAMPLE WEIGHT ON SLUDGE AND ACIDITY DEVELOPMENT

comprehensive study has been made of the oxidation of transformer oils, as measured by the rate of sludge and acidity development, in this region, and the results obtained can be classified as follows :

1. *Variation in Oil Type*

The results for uncatalysed oxidations are summarized in Fig. 2, whilst those for the corresponding catalysed oxidations are shown in Figs. 3 and 4. Graphs are not included for the uncatalysed oxidations of oils, B, C, G, and H, where the rates of sludge and acidity formation are very slow during the periods considered. It may be noted that all the oils, with the exception of oils F and I, are B. 30-class transformer oils meeting B.S. 148 : 1933; oil B meets British, German (V.D.E. 0370/1936), and Swiss (S.E.V. 124/1936) specifications. Oil F is an unrefined spindle distillate (transformer oil feedstock), whilst oil I is the saturates fraction * from a technical white oil.

The following general conclusions are apparent from a consideration of the results obtained :

Five of the oils examined, viz., B, C, E, G, and H, develop very little

* Obtained by a silica gel adsorption fractionation as described by G. H. Beaven, L. F. Foster, and C. N. Thompson. *J. Inst. Petrol.*, 1950, **36** 89.

acidity or sludge over a period of at least twenty-four days in the uncatalysed state. Oxidations of these five oils in the presence of 100 p.p.m. (parts per million) soluble copper indicate that the rates of sludge and acidity development are increased in all cases. However, in the case of

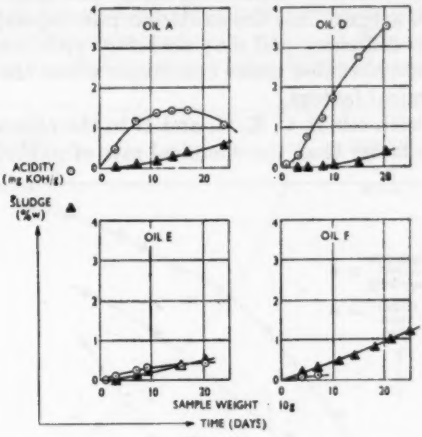

FIG. 2

SLUDGE AND ACIDITY DEVELOPMENT IN VARIOUS TRANSFORMER OILS IN ABSENCE OF CATALYST

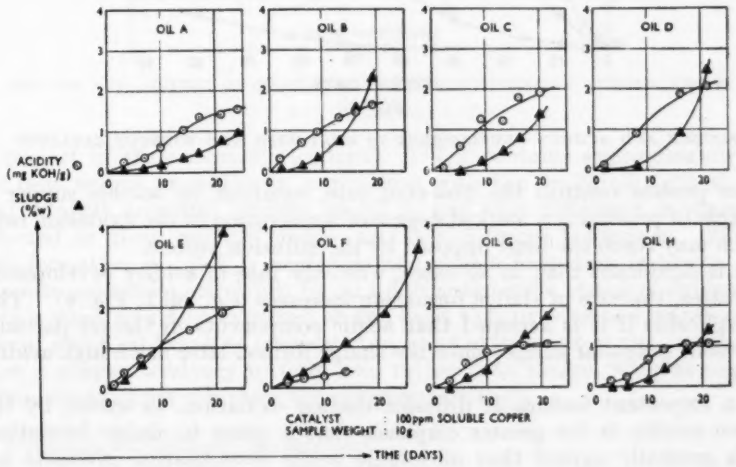

FIG. 3

SLUDGE AND ACIDITY DEVELOPMENT IN VARIOUS TRANSFORMER OILS

the oils developing high acidities in the uncatalysed state (*e.g.*, oils A and D), the rate of acidity development is *decreased* when the oxidation is carried out in the presence of a soluble copper catalyst; the rate of sludge formation is, at the same time, *increased*.

In some cases, *e.g.*, oils A and D, there is no significant degree of acceleration produced in the oxidation reactions by the use of a soluble copper catalyst. Except where the overall rate of oxidation is very slow (*e.g.*, oils B, C, E, G, and H) the oils appear to oxidize with and without catalyst, to very similar total extents, considering sludge and acidity in combination. These facts further suggest that the oxidation rate depends primarily on the physical process of diffusion, and may therefore yield results of a different character from those obtained under conditions where the reaction rates are controlled by chemical factors.

It is clear that with oils B, C, E, G, and H in the absence of catalyst, the rate of diffusion is faster than the chemical rate of oxidation, and since the

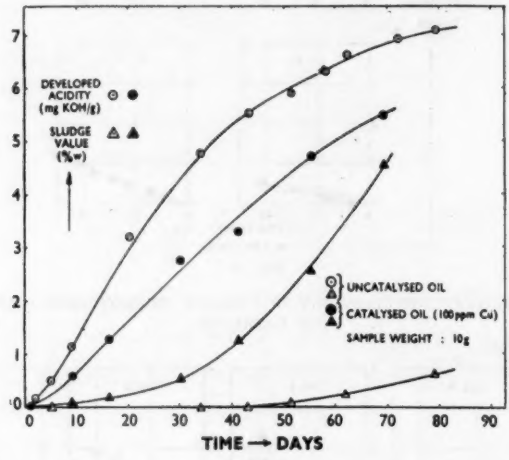

FIG. 4

SLUDGE AND ACIDITY DEVELOPMENT IN OIL I WITH AND WITHOUT CATALYST

latter process controls the observed rate, catalysis by soluble copper is capable of producing a marked degree of acceleration in the oxidation rate, which may reach the limit imposed by the diffusion process.

It is significant that, in all cases, when the rate of acidity development decreases, the rate of sludge formation increases (*e.g.*, oil I, Fig. 4). This is explicable if it is assumed that acidic components in the oil partially represent potential sludge, since the sludge formed later has a high acidity itself.

An important feature of diffusion-limited oxidation, as shown by the above results, is the greater emphasis that is given to sludge formation. It is generally agreed that oil-soluble acidic deterioration products are subsequently converted, at least in part, to oil-insoluble sludge and that this phenomenon is responsible for the fluctuations in acidity observed in extended laboratory oxidation tests and in transformers under service conditions.[10] The overall reduction in oxidation rate resulting from limited oxygen access promotes this slow conversion in comparison with more highly accelerated tests. Fig. 5 summarizes the results obtained for oil C at the same temperature (120° C) and catalyst concentration (100 p.p.m.

soluble copper) as the present studies but under conditions where the rate of deterioration is not limited by oxygen diffusion, the reaction velocity being consequently much higher. It will be seen that, whilst sludge formation tends to be small, acidity development is higher than for the same oil under diffusion-limited conditions (Fig. 3).

The results for oil F, an unrefined spindle distillate (Figs. 2 and 3), and oil I, a saturates fraction from technical white oil (Fig. 4), are interesting in that they represent a study of two widely different products, particularly

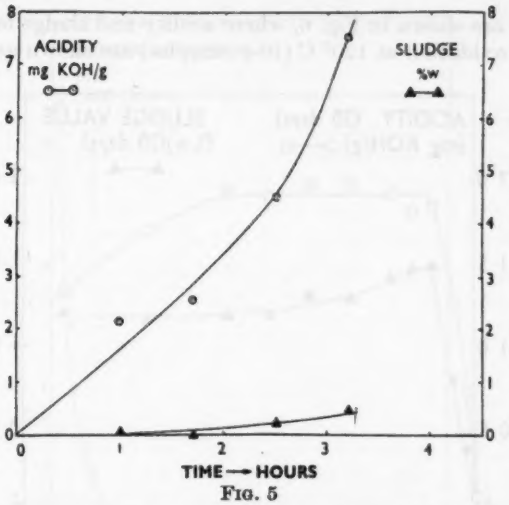

FIG. 5

SLUDGE AND ACIDITY DEVELOPMENT UNDER CONDITIONS OF OXYGEN EXCESS (OIL C)

in respect of their aromatic contents. Oil F contains approximately 50 per cent weight aromatics in comparison with the negligible (<0·02 per cent weight) aromatic content of oil I, and this difference is strongly reflected in their relative sludge- and acid-forming tendencies. Whilst acid formation in oil F is negligible and sludge formation is heavy, the opposite tendencies are shown by oil I during the same period of oxidation. This is what would be expected on the basis that over-refined transformer oils (of which oil I may be considered to be an extreme example), in general show a greater tendency to form acid rather than sludge, whereas under-refined transformer oils show the opposite tendency.

2. *Variation in Catalyst Concentration*

The work described above indicates that, for certain oils which oxidize slowly in the uncatalysed state, the addition of catalyst (100 p.p.m. soluble copper) increases the reaction velocity to such an extent that this then becomes limited by the ease of oxygen access to the oil; this is well shown by oil C, which oxidizes slowly in the absence of catalyst, but forms appreciable amounts of sludge and acidity in the presence of 100 p.p.m. soluble copper. The catalysed rate, however, increases further with a decreasing amount of

reactant oil (Fig. 1). It is clear that, keeping the sample weight and reactant dimensions constant, further increase in the catalyst concentration should, if diffusion at this level is rate-limiting, have little effect upon the rate of deterioration (assuming, of course, a true catalytic action of the copper). It was therefore of interest to study the rate of deterioration over a wide range of catalyst concentrations in order to locate the diffusion-controlled region; clearly, diffusion must become a significant factor under conditions where between zero and 100 p.p.m. soluble copper are present as catalyst.

The results are shown in Fig. 6, where acidity and sludge formation after twenty-days' oxidation at 120° C (10-g samples) are shown as a function of

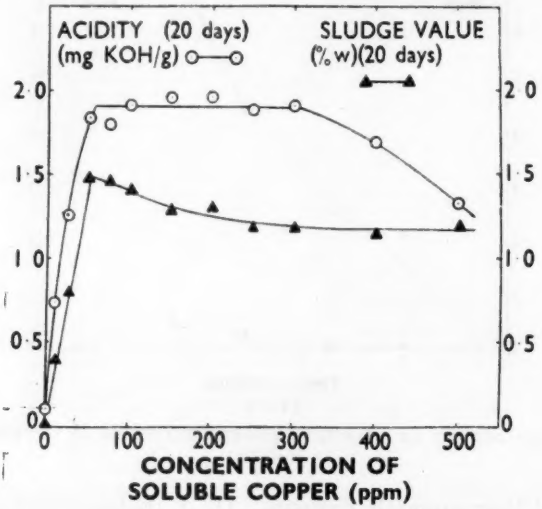

FIG. 6

SLUDGE AND ACIDITY DEVELOPMENT IN OIL C AT VARIOUS CATALYST CONCENTRATIONS

the catalyst concentration from zero to 500 p.p.m. soluble copper. The individual acidity/and sludge/time curves are not shown, as these were of the same general type as that given for oil C in Fig. 3. The significant facts emerging from a study of these data are as follows: the acidity development and sludge formation are critically dependent upon the catalyst concentration in the region of zero to 50 p.p.m.; further increase, however, in the amount of copper present has little or no effect upon the acidity and sludge, which remain constant at *ca* 1·9 mg KOH/g and *ca* 1·3 per cent weight respectively. Only at still higher concentrations (300 to 500 p.p.m.) does any further effect become apparent, the acidity then tending to decrease with increasing amount of catalyst, whilst the sludge formation remains constant.

In order to explain these observations it is necessary to refer to the catalytic response of oil C under conditions where diffusion effects are negligible (*i.e.*, experiments carried out under conditions where oxygen is

present in excess). Fig. 7 summarizes these results, where the time taken (t_{1000}) for 100 g of the oil to absorb 1000 ml of oxygen is related to the concentration (p.p.m.) of soluble copper. From a copper concentration of zero to near 100 p.p.m., the oxygen absorption time decreases steadily (*i.e.*, the oxidation rate increases). With increasing catalyst concentration above this point the oxidation rate remains fairly constant, undergoing minor fluctuations at about 250 p.p.m. copper, and finally decreasing above 400 p.p.m. Similar behaviour has also been reported by George,

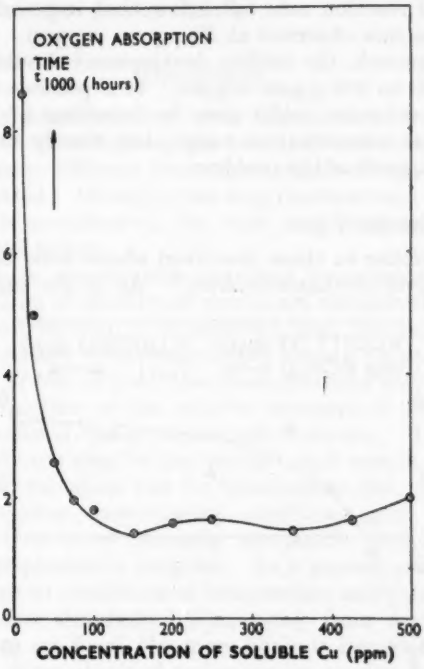

FIG. 7

VARIATION IN REACTION RATE OF OIL C WITH CATALYST CONCENTRATION

Rideal, and Robertson,[11] who used copper and cobalt stearates, in the same concentration range, as catalysts for the oxidation of pure hydrocarbons. This behaviour is similar to that observed under the present oxidation conditions (Fig. 6), with the exception that in the latter case, the acidity and sludge formation, at a fixed time, remain approximately constant at catalyst concentrations greater than 50 p.p.m. Assuming that the function of the catalyst is the same in each case, the only explanation for this difference in behaviour must be that the addition of catalyst has increased the chemical rate of oxidation to such an extent that, at a catalyst concentration of 50 p.p.m., the rate, under the " test-tube " oxidation conditions, approaches that allowed by the diffusion processes. The acidity and sludge remain constant with further increase in the amount of catalyst,

not because the catalyst is no longer functioning in the accepted manner (as is shown by Fig. 7), but because the rate of oxidation has reached the maximum allowed by factors controlling the ease of oxygen access to the oil.

It is not possible, at the moment, to explain in a satisfactory manner, the decrease in acidity development which occurs in the region of 300 to 500 p.p.m. copper, sludge formation at the same time remaining constant (Fig. 6). Although the absolute chemical reaction rate is tending to decrease with increasing catalyst concentration in this region (Fig. 7) the effect is small and, what is more important, at no catalyst concentration does the absolute chemical reaction rate fall below that imposed by the diffusion processes (*i.e.*, the rate observed at 50 p.p.m. copper). Hence, according to this line of approach, the acidity development should remain constant over the range 300 to 500 p.p.m. copper. It is possible that esterification reactions (which consume acids) may be becoming of relatively greater importance in this concentration range, but clearly more work will be necessary on this aspect of the problem.

3. *Variation in Catalyst Type*

Experiments similar to those described above were carried out using a soluble iron catalyst (as naphthenate).* As in the case of catalysis by

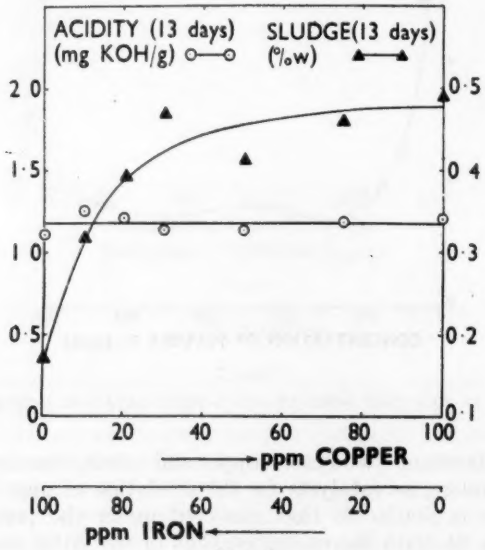

FIG. 8

SLUDGE AND ACIDITY DEVELOPMENT IN OIL C IN THE PRESENCE OF MIXED COPPER/IRON CATALYST

copper, the addition of a soluble iron salt to an oil was found to promote sludge formation preferentially to that of acid. However, at any particular catalyst concentration, iron influenced sludge formation to a lesser

* Prepared in a manner analogous to that used for copper.

extent than did copper, there being little or no difference between the respective acidity developments.

Studies were also carried out using mixed iron–copper catalysts (soluble iron and copper naphthenates). The ratio of copper to iron was varied over a wide range from 100 per cent copper to 100 per cent iron, the total concentration of catalyst being kept constant at 100 p.p.m. Fig. 8 illustrates graphically the results obtained after thirteen days' oxidation at 120° C. It will be seen that whilst acidity development remains constant with increasing ratios of iron to copper, sludge formation tends to decrease, especially for Fe/Cu ratios greater than 4.

Discussion

From the results described, it is clear that, under conditions in which oxygen access to the oil is limited, the reaction rates and product proportions are significantly different from those observed when diffusion phenomena are eliminated. Owing to the long reaction time (*ca* three weeks), sludge formation is accentuated, the slow acid-to-sludge conversion becoming an important factor.

The results assume considerable practical importance when the conditions under which an oil oxidizes in service are considered. As previously mentioned (see Introduction) it is believed that the oxidation reactions occurring in service must be governed very largely by the ease of oxygen access to the oil. If this is true, the present results may be construed to give a qualitative picture of the relative amounts of sludge and acidity which would be produced by the various oils in service. The reaction time, however, is probably too long for the method, as it stands, to be considered suitable as a rapid evaluation test for transformer oils. A test of shorter duration, under similar experimental conditions and yielding similar information, would be more generally acceptable, and investigations in this direction are at present in progress. As a general principle, it may be stated that under given conditions of temperature and catalyst, the further inside the "diffusion threshold" (*i.e.*, away from oxygen excess) the oxidation conditions are fixed, the more likely it becomes that the results will correlate with those obtained under service conditions.

Hurworth [1] has observed that the initial rate of acidity development in a class A transformer oil under service conditions is an exponential function of time, representing an autocatalytic type of growth; after further oxidation, however, the acidity/time curve assumes a linear form, the rate of increase of acidity being constant, and independent of time.

Hurworth believes that this change in the acidity/time curve from autocatalytic to linear form may be caused by a shortage of oxygen resulting from "friction losses" which increase as the autocatalytic oxidation proceeds. This process he states—

> "continues until nearly all the atmospheric pressure of oxygen is utilized in overcoming friction losses. The maximum amount of oxygen is now entering the transformer, and the rate of oxidation has reached a peak value which is associated with the linear part of the acid characteristic curve."

A simpler explanation has been advanced by Shaw,[12] who suggests that as the acid concentration increases, the rate of evaporation of volatile acidic products also increases until there is equilibrium between the rates of acid generation and evaporation.

It is to be noted that sludge formation is not considered in either of the above theories, and it would appear from the results of the present work that the formation of this reaction product will also contribute to the effect described by Hurworth. The conditions are such that the rate of oxidation is relatively slow, thus favouring the slow acid-to-sludge conversion; furthermore, the change in form of the acidity/time curve occurs at an acidity of *ca* 1·0 mg KOH/g, that is, at a value where a significant amount of copper may be dissolved in the oil and thus sludge formation will again be favoured (cf. Fig. 4). It has been shown above that the onset of sludge formation, either in class A or B oils, is always associated with a decrease in the rate of acid development, and it is thus suggested that this phenomenon will also account for Hurworth's observation.

Conclusions

1. Under conditions in which oxygen access to the oil is limited, the reaction rates and product proportions are significantly different from those observed when diffusion factors are eliminated.
2. For oils which show very low rates of acid and sludge formation during uncatalysed oxidations, the presence of a soluble catalyst markedly accelerates the reaction rate.
3. Catalysis by oil-soluble copper or iron in the case of other oils has little or no effect upon the rate of oxidation, but may alter the relative proportions of acid and sludge formed. In such cases diffusion is a rate-limiting factor in the oxidations. In general, oil-soluble catalysts promote sludge formation preferentially to that of acid.
4. In all cases where the rate of acidity development decreases, the rate of sludge formation increases.
5. The overall reduction in oxidation rate, resulting from limited oxygen access, promotes a slow acid-to-sludge conversion in comparison with more highly accelerated oxidations.
6. The oxidation rate is critically dependent upon the catalyst concentration up to a limiting value which is determined by the rate of oxygen diffusion into the oil. As would be expected, further increase in catalyst concentration has relatively little effect upon the oxidation rate as measured by acidity development.
7. A soluble iron catalyst influences sludges formation to a lesser extent than does copper.

Acknowledgments

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ALKYLNAPHTHALENES. PART VI. 1 : 3-DIMETHYLNAPHTHALENE

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The preparation and the physical properties of the ten dimethylnaphthalenes have been described in Part I [1] of this series. Several of these hydrocarbons were obtained *via* five-stage syntheses which yielded only 1 to 2 g of the final product, insufficient for thorough purification and examination. 1 : 3-Dimethylnaphthalene was one case in which it seemed advisable to repeat the preparation on a scale sufficient to yield 100 g for the final purification.

The synthesis was an elaboration of that by Barnett and Sanders,[2] starting from highly purified *m*-xylene and building on the second ring by the succinic anhydride method. By the use of different reagents at some of the stages far better yields than those of Barnett and Sanders, or those of Part I, were obtained. Intermediates were all carefully purified, and the final product was subjected to a series of purifications by distillation and by low-temperature crystallization, the physical constants being determined after each process.

It was thought that the highly crystalline 5 : 7-dimethyltetralone (5 : 7-dimethyl-1-keto-1 : 2 : 3 : 4-tetrahydronaphthalene) of convenient melting point (49°) would be the best guarantee for the purity of the (liquid) 1 : 3-dimethylnaphthalene, but surprisingly, at the penultimate stage of the synthesis the dimethyltetrahydronaphthalene crystallized readily at −7° and could be recrystallized from acetone. Thus, in this synthesis of 1 : 3-dimethylnaphthalene a crystalline, sharp-melting solid has been obtained at every stage; the overall yield from *m*-xylene averaged 52 per cent.

The final values of the melting point, −4·17°, and of the refractive index, n_D^{20} 1·6100, are both higher than those recorded in Part I.[1] As to the discrepancy in the literature regarding the styphnate, the value here obtained for the melting point (116°) supports that of Kloetzel [3] (116° to 118°). Jitkow and Bogert [4] recorded m.p. 132° to 133° for a specimen recrystallized from *aqueous* alcohol.

Experimental

m-*Xylene*. A specimen of b.p. 137° to 141°/760 mm and n_D^{20} 1·4940 was sulphonated according to the directions of Mair *et al.*[5] The resulting mixture of sulphonic acids was kept at 130° to 140° (thermometer in the acid) while superheated steam was passed through the melt; the hydrocarbon which distilled had f.p. −47° to −50°, n_D^{20} 1·4976. Sulphonation of this distillate gave brownish crystals which were crystallized from chloroform until colourless and of constant m.p. 62° to 63°. Patterson [6] gave m.p. 61° to 62°. Titration of these crystals with alkali showed the

equivalent to be 224·9 ($C_6H_3Me_2{\cdot}SO_3H{\cdot}2H_2O$ requires 222·1). On desulphonation at 130° to 140° this acid yielded hydrocarbon of n_D^{20} 1·4976. In order to make certain of the absence of sulphur compounds the specimen was treated with mercuric acetate following the directions given by Dimroth [7] for benzene. 400 cc of the xylene and a solution of mercuric oxide (16 g) in acetic acid (16 cc) and water (120 cc) were heated under reflux for 8 hr. After separation and distillation of the hydrocarbon layer a small amount of yellow solid remained. Fractionation of the xylene with a Stedman column gave a large main fraction of b.p. 138·8°/758·8 mm, n_D^{20} 1·4971, D_4^{20} 0·8642, and f.p. −48°; this was used for the

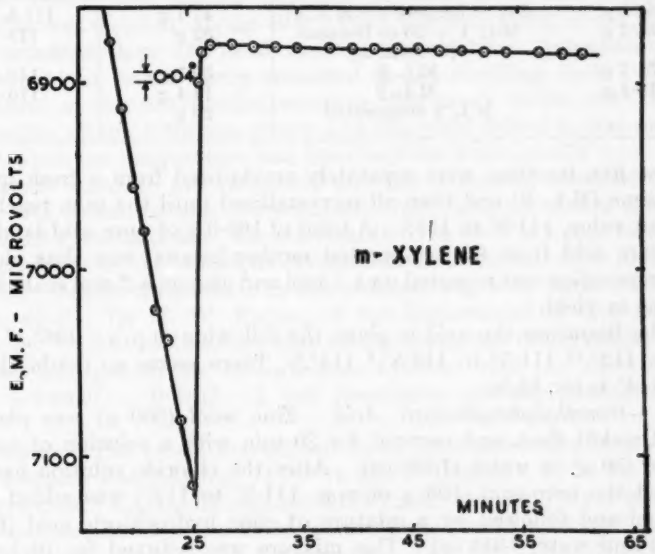

FIG. 1

synthesis. The accepted values for the constants are : [8, 9] b.p. 139·10°/760 mm; n_D^{20} 1·4972; D_4^{20} 0·86417; f.p. −47·872°.

For calibration of a thermocouple some of the best *m*-xylene was further purified. It was slowly cooled in ethanol–carbon dioxide until three-quarters had frozen; the liquid was drained off and the solid then melted. Repetition of the process gave a specimen of n_D^{20} 1·4970; D_4^{20} 0·8642. The behaviour on freezing indicated a purity of 99·94 moles per cent. (Fig. 1.)

β-2 : 4-*Dimethylbenzoylpropionic Acid*. Powdered aluminium chloride (255 g, 1·91 mol) was added with swirling to a mixture of succinic anhydride (92·5 g, 0·925 mol) and pure *m*-xylene (125 cc, 1·03 mol) in tetrachloroethane (350 cc, recently distilled). Evolution of hydrogen chloride was almost immediate, the liquid turning deep red and becoming warm. After standing for 36 hr the mixture was decomposed by addition of ice and conc hydrochloric acid (150 cc). The tetrachloroethane and unchanged xylene were removed by steam-distillation, the residue was poured into beakers and cooled to 0°. When the brown solid which separated had been collected

on a filter it was extracted three or four times by boiling under reflux with (each time) 500 cc of 10 per cent sodium carbonate solution. The carbonate extracts were boiled with charcoal, filtered, and carefully acidified with hydrochloric acid. After drying in air the resulting colourless solid acid weighed 186 g (97·5 per cent yield calculated on the succinic anhydride) and melted at 108·5 to 111·5°.

Recrystallized in portions from benzene the crude product yielded pure acid of m.p. 111·5° to 112° :—

Wt. crude acid	Solvent	Wt. product	M.p., ° C
60·0 g	200 cc benzene (= M.L. 1)	47·1 g	111·5–112
64·2 g	M.L. 1 + 50 cc benzene (= M.L. 2)	53 g	111–112
51·7 g	M.L. 2	50·7 g	110–111
10·2 g	M.L. 2	14·4 g	110–112
	M.L. 2 evaporated	20 g	

These five fractions were separately crystallized from a fresh quantity of benzene (M.L. 3) and then all recrystallized until the m.p. reached the constant value, 111·5° to 112°. A total of 160·9 g of pure acid (and 16·7 g of impure acid from the evaporated mother-liquors) was thus obtained. This preparation was repeated on a 1-mol and also on a 2-mol scale without decrease in yield.

In the literature the acid is given the following m.p.'s : 106°,[10] 108°,[11] 111° to 112°,[12] 111·5° to 112·5°,[1] 114°.[2] There seems no doubt that the value 114° is too high.

γ-2 : 4-*Dimethylphenylbutyric Acid*. Zinc wool (300 g) was placed in a 2-l. Quickfit flask and covered for 30 min with a solution of mercuric chloride (50 g) in water (1500 cc). After the chloride solution had been decanted the keto-acid (100 g of m.p. 111·5° to 112°) was added to the zinc wool and followed by a mixture of conc hydrochloric acid (600 cc, D 1·16) and water (400 cc). This mixture was refluxed for 10 hr, conc hydrochloric acid (40 cc) being added each hour. Finally, 100 cc of conc acid were added and the refluxing continued overnight : almost all the zinc dissolved, leaving a globule of mercury. The hot contents of the flask were poured into a beaker and then cooled with ice-water. The clean, white crust which formed was broken up, washed well on a Buchner filter, and left some days in the air. In several experiments the yield varied from 90 to 93 g (almost quantitative) and the acid melted at 74° to 76°.

The acid was systematically crystallized (compare the keto-acid, above) using benzene–light petroleum (b.p. 60° to 80°) in preference to the acetic acid used in Part I, and the final value of the m.p. was 76·3° to 77·3°. In the literature the values given are : 70°,[13, 14] 71°,[12] 74°,[15, 16] 76° to 76·5°,[17] 76·5° to 77°,[1] 78° to 79°,[18] 79°.[2]

5 : 7-*Dimethyl*-1-*keto*-1 : 2 : 3 : 4-*tetrahydronaphthalene*. For the ring-closure phosphoric acid is much preferable to sulphuric acid.[19, 20]

Phosphorus pentoxide (240 g) was added gradually with continuous stirring (using a thermometer) to orthophosphoric acid (200 cc, D 1·75) in an 800-cc beaker. The temperature rose to 170° to 175°, and a liquid

containing a few viscous lumps was obtained. When the temperature fell to 160° powdered dimethylphenylbutyric acid (60 g) was added; this formed a colourless layer over the dehydrating mixture (which had become dark-brown) and caused the temperature to fall to 140°. The mixture was strongly heated with constant stirring and, at 150° to 155°, the two layers merged to a viscous, green liquid. After having been kept at 160° for 3 min (not longer) the hot mixture was stirred into a beaker of cold water. Before the mixture was quite cold ether was added and the liquids were transferred to a separating-funnel, where, in all, three ether extractions were carried out. After the combined red-brown ethereal extracts had been washed with sodium carbonate solution, dried, and evaporated, the residue was distilled, giving 49·8 g (92·1 per cent yield) of the slightly yellow tetralone (b.p. 113°/0·05 mm) which solidified immediately. A small amount of brown syrup remained in the distilling flask. [If the temperature of the dehydrating mixture rises much above 160° further condensation of the tetralone occurs and the yield falls: it was only 74 per cent when the temperature had been kept for 3 min at 174°.]

The crude tetralone was redistilled and the colourless, constant-boiling fraction was systematically recrystallized from light petroleum (b.p. 60° to 80°) until a constant m.p. was reached, 48·5° to 49·5° in a capillary tube. Some specimens of this tetralone crystallized in beautiful, clear rods, 2 to 5 cm long; others crystallized in fine needles, but both forms melted at 48·5° to 49·5°. Dr M. W. Porter, of the Department of Geology and Mineralogy, Oxford University, who kindly examined the crystals, found that they belonged to the orthorhombic system, with the axial ratios: $a : b : c = 0{\cdot}6946 : 1 : 0{\cdot}3466$. A full description will be published elsewhere.

Derivatives. The *oxime* crystallized from light petroleum (b.p. 60° to 80°) in feathery needles, m.p. 147·5° to 148°. (Found: C, 76·3; H, 8·1; N, 7·3 per cent. $C_{12}H_{15}ON$ requires C, 76·1; H, 8·0; N, 7·4 per cent.)

The semicarbazone, crystallized from ethanol, melted at 239° to 240° (corr). (Found: C, 67·1; H, 7·4 per cent. Calc for $C_{13}H_{17}ON_3$, C, 67·5; H, 7·4 per cent.) In the literature the m.p's given are 234° to 235°,[12] 243° to 246° (decomp).[18] The *phenylhydrazone*, slowly heated, decomposed at about 114°; but when rapidly heated it melted at 130° to 131° (decomp). It was readily oxidized in the air. (Found: C, 81·7; H, 7·8; N, 10·5 per cent. $C_{18}H_{20}N_2$ requires C, 81·8; H, 7·6; N, 10·6 per cent.)

The 2 : 4-dinitrophenylhydrazone crystallized from ethanol in small crimson needles and melted at 273° to 274° (corr). Tucker *et al* [16] give m.p. 270° to 271°. (Found: C, 61·2; H, 5·0; N, 15·9 per cent. Calc. for $C_{18}H_{18}O_4N_4$, C, 61·0; H, 5·1; N, 15·8 per cent.)

In the literature this tetralone, 5 : 7-dimethyl-1-keto-1 : 2 : 3 : 4-tetrahydronaphthalene, is reported as having been prepared by various methods involving ring-closure. (See Table on p. 84.)

The syrup produced as a by-product solidified on shaking with ether. The solid, m.p. 172° to 181°, was separated into two fractions, (*a*) soluble, and (*b*) sparingly soluble, in boiling ethyl alcohol. (*a*) After one recrystallization from light petroleum and two from ethanolic benzene, formed a pale yellow powder, m.p. 161° to 164° (uncorr). (*b*) After six crystallizations from ethanolic benzene formed a white solid, m.p. 195° (uncorr).

Method	Yield per cent	B.p., °C	M.p., °C
Conc H_2SO_4 . . .	10–15	180–182°/17 mm	49–50 [12]
Acid chloride + $AlCl_3$.	40	,,	,, [12]
,, ,, .	92·5	160°/14 mm	liquid [13]
,, ,, .	85–90	159°/12 mm	*ca* 40 [15]
,, ,, .	62	—	46 [16]
Conc H_2SO_4 . . .	30	—	50 [2]
,, . . .	60	130°/0·4 mm	47·5–49 [1]
$H_3PO_4 + P_2O_5$ . .	92	113°/0·25 mm	48·5–49·5 (This paper)

(*a*) Found: C, 92·16; H, 7·77 per cent. M.wt. in camphor, 299. (*b*) Found: C, 92·58; H, 7·56 per cent. M.wt. in camphor, 272. $C_{24}H_{24}$ (or $2C_{12}H_{14}O–2H_2O$) requires C, 92·3; H, 7·77 per cent, M.wt., 312. These condensation products are being further investigated.

5 : 7-*Dimethyl*-1 : 2 : 3 : 4-*tetrahydronaphthalene*. Following the method of Huang-Minlon,[21] the tetralone was reduced by heating its hydrazone with a solution of sodium hydroxide in either ethylene glycol or diethylene glycol.

The pure tetralone (17·4 g, 0·1 mol), hydrazine hydrate (16 cc of 90 per cent, 0·3 mol) and a solution of sodium hydroxide (14 g, 0·35 mol) in ethylene glycol (220 cc) were heated under reflux at *ca* 150° for 1½ hr. The mixture was then distilled with a thermometer in the *liquid* (and the distillate collected) until the boiling point reached 198°; the condenser was fitted and refluxing continued for 5 hr at 198°.

After cooling, the reaction mixture and also the distillate up to 198° were extracted with light petroleum (b.p. 60° to 80°); the extracts were washed with 20 per cent hydrochloric acid, with water and then dried with sodium sulphate. Evaporation of the solvent and distillation of the residue gave 14·8 g (92 per cent yield) of colourless dimethyltetralin, b.p. 126° to 127°/17 mm, and some yellow solid was left in the flask. This solid, after two crystallizations from ethanol–benzene and one from benzene, had m.p. 218° to 219°. (Found: C, 83·9; H, 8·0; N, 7·9 per cent. The azine, $C_{24}H_{28}N_2$, requires C, 83·7; H, 8·2; N, 8·1 per cent.)

Similarly 157 g of the tetralone were reduced in small portions (some with diethylene glycol as solvent) giving 132 g (92 per cent yield) of the tetralin. Distillation of the product with a short column gave (*a*) a small fraction, b.p. up to 251·8°/752 mm, n_D^{20} 1·5388, and (*b*) the main fraction, b.p. 251·8° to 252·4°, n_D^{20} 1·5400.

The main fraction crystallized easily from acetone below −15° and was collected on the filter described by Smith.[22] 10 g of this fraction in redistilled acetone (70 cc), cooled to −70°, gave 8·7 g (after removal of solvent *in vacuo*) of n_D^{20} 1·5399 and m.p. −6·7° (thermometer in the liquid). The 8·7 g, recrystallized from acetone at −60°, gave 6·3 g of n_D^{20} 1·5400, m.p. −6·7°, D_4^{20} 0·9583; $[R_L]_D^{20} = 52·48$. As the calculated $[R_L]_D^{20}$ for $C_{12}H_{16}$ is 51·82 the Exaltation is 0·66.

By a combination of distillation and low-temperature crystallization the bulk of the dimethyltetralin was purified to the constant values, b.p. 253·1°/760 mm; n_D^{20} 1·5399; D_4^{20} 0·9583; m.p. −6·7° (Fig. 2). Krollpfeiffer [12] recorded b.p. 250° to 252°/760 mm; n_D^{20} 1·541; D_4^{20} 0·960. In Part I the values were b.p. 252°/742 mm; n_D^{20} 1·539.

1 : 3-*Dimethylnaphthalene.* The tetralin was dehydrogenated by refluxing with palladium–charcoal catalyst (10 per cent Pd).

Preparation of the dehydrogenating catalyst. 4–5 g of good-quality charcoal were heated ½ hr at 12 mm on the water-bath in a 50-cc flask fitted with a dropping funnel. A solution of palladous chloride (1 g) in water (30 cc) with a few drops of hydrochloric acid was run on to the charcoal, the air admitted and the mixture then transferred to the hydrogenator bottle with the aid of more water. The palladium chloride was reduced by shaking with hydrogen at 2 atm for ½ hr, the theoretical amount of hydrogen being absorbed in 10 min.

After the mixture had been filtered on a Buchner funnel the charcoal was washed with water until free from chloride ($AgNO_3$ test), and dried in a desiccator at 0·1 mm over calcium chloride for 12 hr. Finally, the palladium–charcoal was heated in a drying pistol (P_2O_5) while the pressure

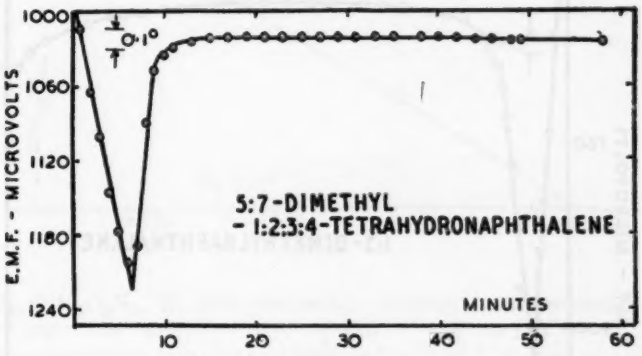

FIG. 2

was *gradually* reduced to 15 mm and the temperature raised to 290°. Catalysts prepared in this way will rapidly dehydrogenate tetrahydrocarbazole (a useful test-substance) at 240°. It is best to keep the catalyst in the drying-pistol until all has been used.

Dehydrogenation. The tetralin (12·7 g) and the catalyst (1 g) were placed in a 50-cc Pyrex Claisen flask fitted with an inlet-tube for nitrogen, a thermometer, and a rubber extension of the side-tube dipping just under the surface of alcohol in a small beaker, so that the bubbles of gas could be counted. The flask was protected from draughts and heated in a metal-bath. At about 240° the liquid evolved hydrogen copiously and the bath-temperature was adjusted to keep the liquid refluxing. After 1 hr little hydrogen was being evolved, but it was advisable to complete the reaction; after 3 hr a slow stream of nitrogen was passed through to sweep out the hydrogen.

Occasionally the nitrogen was shut off and the bubbles of gas counted at the exit. When no more hydrogen was detectable the flask was cooled and the contents extracted with ether. (This was preferable to distilling away from the catalyst.) To the ethereal extract the alcohol from the bubble-counter was added Evaporation of the solvents and distillation

of the residue gave 11·8 g (95·3 per cent yield) of colourless dimethylnaphthalene, b.p. 125° to 140°/12 mm. The high refractive index, n_D^{20} 1·6098, showed that dehydrogenation was almost complete. Up to 40 g of the tetralin at a time were dehydrogenated in this way, and a total of 122·6 g of tetralin gave 112·8 g of the naphthalene, a yield of 94·4 per cent. This product, n_D^{20} 1·6090, was quite stable to dilute alkaline permanganate solution.

Fractional distillation with a column still showed that the dehydrogenation product was not quite homogeneous, the fore-runnings boiling below 140°/22 mm having low refractive indices, 1·605 to 1·607. Crystallization of the better fractions, n_D^{20} above 1·6090, from acetone (12 g of hydrocarbon to 45 cc of acetone) at −70° raised the n_D^{20} to 1·6099, but failed to improve

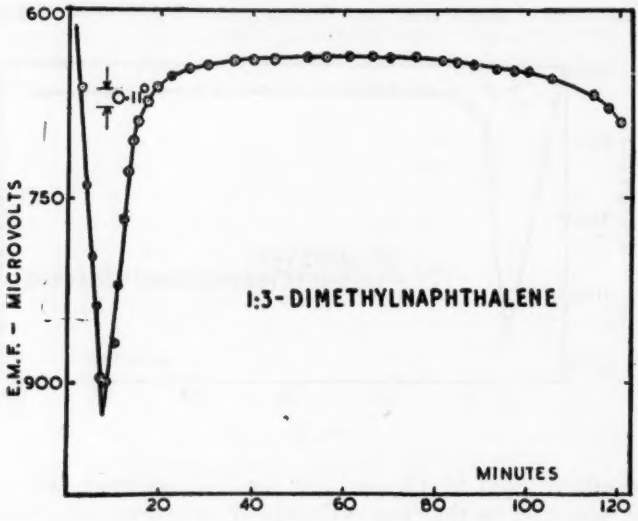

FIG. 3

the fore-runnings. Better results were obtained with light petroleum (b.p. 40° to 60°, freed from olefins): taking first 12·2 g of n_D^{20} 1·6100 in 60 cc of light petroleum and cooling to −72° gave 6·4 g of n_D^{20} 1·6100; then several specimens with n_D^{20} as low as 1·6094, on passing through this mother-liquor, gave products of n_D^{20} 1·6100; finally, a specimen of n_D^{20} 1·6090 was improved to n_D^{20} 1·6096.

Fractional distillation at atmospheric pressure did not yield satisfactory products, as decomposition slowly occurred at the high temperature of the boiling point. A copper–constantan thermocouple in the still-head indicated that the b.p. was 265·0° ± 0·1° (Fig. 4).

For the best specimen of 1 : 3-dimethylnaphthalene the constants were: b.p. 265·0° ± 0·1°; f.p. (determined on a 40-g sample in the Rossini apparatus)[23] −4·17° (Fig. 3); n_D^{20} 1·6100, on a Zeiss–Abbé refractometer; D_4^{20} 1·0063. From these data the molecular refraction, $[R_L]_D^{20}$, is 53·85; the Exaltation is thus 2·97.

Molecular Compounds. The picrate crystallized from ethanol in orange needles, m.p. 116° to 117°. (Found : N, 11·2 per cent. Calc. for $C_{12}H_{12}{\cdot}C_6H_3O_7N_3$, N, 10·9 per cent.) Melting points recorded in the literature are : 58° to 59°,[24] 115° to 117°,[16] 116° to 117°,[1, 4] 117° to 118°,[3] 118°.[2]

The yellow styphnate, prepared in ethanol and recrystallized from ethanol, melted at 115° to 116·5°. (Found : N, 10·65 per cent. Calc.

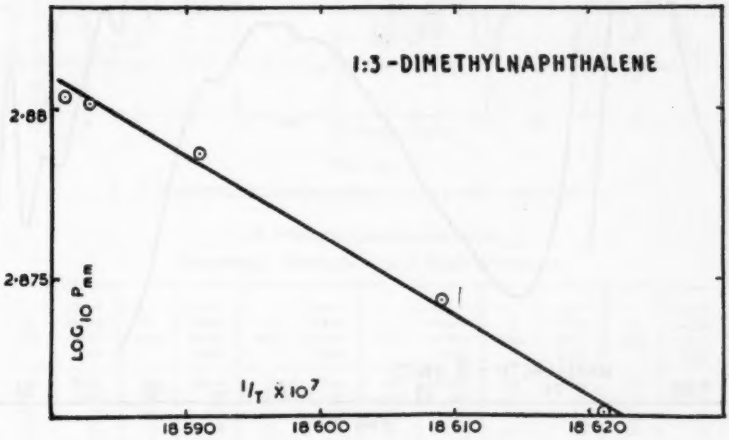

FIG. 4

for $C_{12}H_{12}{\cdot}C_6H_3O_8N_3$, N, 10·5 per cent.) Melting points recorded in the literature are : 116° to 117°,[1] 116° to 118°,[3] 132° to 133°.[4]

The trinitrobenzene complex, as described in Part I, melted at 135°.

Samples of pure 1 : 3-dimethylnaphthalene have been sent to a number of spectroscopists. The authors are grateful to the directors of the Koninklijke/Shell Laboratorium, Amsterdam, for permission to publish the following data on the infra-red and the ultra-violet spectra ; they wish also to thank Mr K. van Nes and Dr J. Beintema for their co-operation.

Ultra-violet	1 : 3-Dimethylnaphthalene
Band (Å)	log ε
3222	2·677
3168–3170	2·498
3078	2·785
2915	3·656
2835	3·762
2805	3·763
2755	3·732
2660	3·591
2285	4·980
about 2250	4·856

Solution in methyl*cyclo*hexane ; temp. 25° ; cell length, 1·00 cm. Concentration (g per 100 c.c.) : (i) 0·0000977, (ii) 0·000977, (iii) 0·00977.

These spectrographic data replace those recorded for a 97 per cent pure

specimen of 1 : 3-dimethylnaphthalene, A.P.I. Serial Nos. 216 and 767 (1948), compare Parts I [1] and IV [25] of this Series.

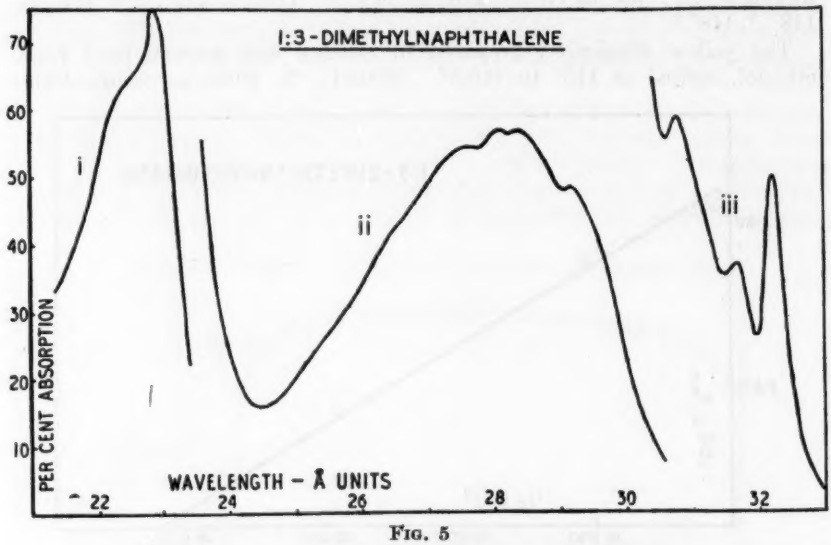

FIG. 5

1 : 3-DIMETHYLNAPHTHALENE (ULTRA-VIOLET SPECTRUM)

1 : 3-DIMETHYLNAPHTHALENE

Table of Frequencies of Infra-red Absorption Maxima

Frequency (cm^{-1})	Frequency (cm^{-1})
748	1278
775	1351 (s)
797 (s) *	1378
845	1410
862	1443
890	1464 (s)
921	1517
949	1550
983	1585 (s)
1011 (s)	1605
1032	1628
1070	1696
1137	1724 (s)
1163	1745
1180	1795
1218	1910 (s)
1236	1924

* s signifies "shoulder."

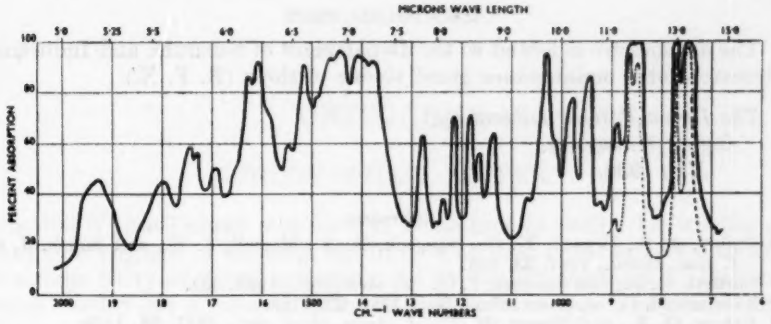

FIG. 6

1 : 3-DIMETHYLNAPHTHALENE (INFRA-RED SPECTRUM)

1 : 3-DIMETHYLNAPHTHALENE

Percentage Absorption and Wave Numbers

% abs.	cm.⁻¹ wave numbers	% abs.	cm.⁻¹ wave numbers	% abs.	cm.⁻¹ wave numbers	% abs.	cm.⁻¹ wave numbers	% abs.	cm.⁻¹ wave numbers	% abs.	cm.⁻¹ wave numbers
11	2012	87	1616	64	1351	66	1048	39	789	39	875
12	1988	92	05	49	37	95	32	43	86	60	71
23	72	90	03	38	32	83	27	62	81	83	67
40	57	79	1595	31	24	61	18	82	79	93	65
44	38	73	90	30	16	61	11	100	75	95	62
45	24	71	85	28	09	47	02	100	73	84	57
42	19	70	82	27	02	58	992	100	71	83	55
39	12	62	77	29	1295	74	83	100	70	89	52
37	05	55	72	34	90	51	75	83	67	93	45
35	01	50	63	48	87	45	69	83	62	87	45
29	1894	49	60	63	78	62	60	91	58	76	43
25	1890	52	55	48	71	86	49	97	56	41	38
20	76	59	50	39	67	68	43	100	52	24	33
18	66	58	46	30	61	38	38	100	48	18	28
23	52	56	41	27	56	35	35	100	45	15	20
32	35	66	31	28	51	36	29	100	43	15	15
34	28	71	27	28	45	36	21	99	42	15	10
44	05	83	22	38	36	35	20	99	40	15	03
44	1795	91	17	34	35	33	17	91	36	16	797
42	92	89	11	30	30	34	10	80	33	17	89
39	86	77	02	37	25	37	05	64	30	20	86
36	79	74	1499	70	18	61	896	50	28	30	81
35	70	77	93	52	14	68	90	43	24	47	79
46	61	78	88	35	09	62	87	38	22	87	75
56	54	81	84	30	03	59	83	36	19	95	75
58	45	85	77	30	1199	75	75	35	17	85	71
53	39	93	64	31	93	91	71	32	12	75	70
46	30	93	56	35	89	99	67	30	09	47	67
45	24	95	49	48	85	99	65	30	07	40	62
42	21	96	43	71	80	99	63	28	02	52	58
41	12	95	39	57	75	100	57	27	697	71	56
47	04	93	35	45	71	100	55	27	93	92	52
49	1698	83	25	56	63	100	52	25	91	99	48
49	96	86	18	38	53	100	49	26	88	99	45
49	92	93	14	44	45	100	45	26	83	97	43
45	86	94	10	64	37	100	43	26	80	94	42
38	75	93	04	33	26	84	38			82	40
40	69	92	1397	25	13	57	33			56	36
44	61	90	87	22	01	46	28			40	33
54	47	92	81	24	1089	33	20			29	30
63	42	92	78	28	83	31	15	27	896	23	28
77	37	87	70	38	70	30	10	30	90	21	24
86	28	75	64	37	64	31	03	26	87	26	22
83	23	66	59	44	57	37	797	25	83	19	19

State: Liquid Temperature: 25° C. Cell Length: 0·1096 mm, 0·028 mm.

ACKNOWLEDGMENT

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The Dyson Perrins Laboratory,
Oxford University.
May 1950.

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OBITUARY

GORDON WILLIS LEPPER

GORDON WILLIS LEPPER was born at Folkestone in 1889. He was for a short time engaged in teaching, but in 1909 he was chosen to be a member of a large party of young geologists, led by the late Leonard V. Dalton, which carried out a reconnaissance in Venezuela in search of petroleum. The late Mr Cunningham Craig met the party in Trinidad and was impressed by Lepper, and this led to an appointment on the geological staff of the Burmah Oil Company. From 1910 until 1921 Lepper was engaged mainly in geological exploration in Burma and India, and proved himself to be a keen, conscientious, and exact worker. After study leave in London to complete his Hons. B.Sc. course at the Imperial College he returned to Burma, and was the company's senior geologist in the East until he retired in 1936. During this period many new members joined the geological staff and were encouraged and helped under Lepper's kindly leadership. After returning to England he became geological adviser to the Petroleum Division of the Ministry of Fuel and Power until 1946, when he returned to the Burmah Oil Company.

Lepper became an Associate Member of the Institute in 1914, a Member in 1920, and a Fellow in 1939 when the Fellowship was instituted. He had a wide experience of the varied applications of geology to petroleum exploration and development, and contributed to discussions on these subjects. He also had a very detailed knowledge of the geology of Burma and the adjacent parts of India, and contributed an important article on the geology of the oil-bearing regions of Burma and of Assam–Arakan to the first World Petroleum Congress in 1933. In November 1938 Lepper gave a lecture to the Institute at the House of the Royal Geographical Society on "The Search for Oil in Britain." The lecture was illustrated by exhibits and a film, and was so much appreciated that it was repeated at the Northern, Birmingham (Student's), and South Wales branches of the Institute. He was also a Fellow of the Geological Society of London from 1914 until his death in September 1950.

Lepper married in 1913, and his wife died in 1929. He married again in 1930, and leaves a widow, three daughters, and two sons.

T. D.

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Oilfield Exploration and Exploitation

Geology

287. Oilfield fossils of the Lower Eocene. D. T. Setterington. *Petroleum*, Aug. 1949, **12** (8), 197–9.—Describes the important genera and species of foraminifera, lamellibranchia, gastropoda, and echinoidea of the Lower Eocene, all of which are found useful in stratigraphical work. R. E. P.

288. Are oil shales natural source beds of petroleum? Part 1. B. J. Ferris. *World Oil*, Aug. 1950, **131** (3), 80.—Coal, cannel coal, marine black shale, oil shale, limestone have each in turn been suggested as petroleum-source rocks. Experiments have been carried out on Green River shale samples to investigate the possibility that oil shales are source beds.

Organic constituents of oil shales are of two main types: an amorphous brown isotropic substance, and fragments of organisms imbedded in the "kerogen." Microscopic and solubility studies of the kerogen have shown that its solubility is inversely proportional to particle size and sp. gr., and proportional to the time exposure of the solvent and its temp, and oil yield. C. A. F.

239. Are oil shales natural source beds of petroleum? Part 2. B. J. Ferris. *World Oil*, Oct. 1950, **131** (5), 85.—Oil shales have been considered to be important petroleum sources, since they generate oil when heated, are rich in organic remains, and were formed under reducing conditions in shallow seas.

There is much evidence that most of the world's oil was generated below the temp required for the destructive distillation of oil shales. Oil may have been generated from shales over long geological periods at low temp.

Samples of Estonian and Green River oil shales were heated for seven days and extracted at four different temp. It was found that the greatest increase in percentage soluble extract was at 340° C, the highest temp to which the samples were heated. During twenty-eight days' heating at subconversion temp no free oil was formed. When the shales were mixed with barium chloride and copper, to act as catalysts, larger extracts were obtained. No increase in extract was obtained when radioactive minerals were mixed with the shale.

As a result of these experiments it is concluded that oil shales have not been a significant source of petroleum.

Fourteen references are given. C. A. F.

290. Arkansas field proved. Anon. *Petrol. Engr*, Nov. 1950, **22** (12), B-86.—1 Concannon, on the Columbia–Union County border, Arkansas, on test produced 79·75 b.d. distillate from the Smackover at 9286–9290 ft. G.O.R. was 9850 : 1. On a test of the overlying Cotton Valley, from 8024 to 8044 ft, the well produced 144 b.d. of 46° oil. G.O.R. was 450 : 1. The well has been dually completed. C. A. F.

291. New Bishop well in Cuyama. Anon. *Petrol. Engr*, Oct. 1950, **22** (11), B-96.—Bishop Hancock 44–31, in the Cuyama area, California, on an 8-hr test pumped 510 b.d. The well is 2½ miles from the nearest producing field. C. A. F.

292. Rank wildcat opens Colorado oil pool. Anon. *Petrol. Engr*, Oct. 1950, **22** (11), B-99.—1 Hanson, 20 miles northeast of the Greasewood pool, Colorado, on a 2-hr test flowed gas in 5 min and had 4400 ft of 38° oil in the hole. Pay was Muddy Sand, topped between 6715 and 6726 ft. C. A. F.

293. Wildcatting in Florida. J. E. Banks. *Oil Gas J.*, 22.6.50, **49** (7), 252.—Geologically Florida belongs to the Gulf Coast Cretaceous oil and gas area. In North and South Florida the Cretaceous section is believed to exceed 10,000 ft, but on the Ocala uplift it is only 1500 ft. The Cretaceous is masked by thick Tertiary limestone. At Sunniland production is obtained from a thin limestone of Glen Rose age. Drilling is difficult in the Tertiary deposits because of lost circulation. The difficulties of drilling are discussed in some detail. Cave-ins are common; stuck pipe is frequent. Apart from initial cavities the hole may enlarge in places during drilling. Wells become crooked.

A table gives costs for a number of wildcats. G. D. H.

294. Illinois area opened. Anon. *Petrol. Engr*, Nov. 1950, **22** (12), B-90.—1 Wells, Richland County, Illinois, on a 1-hr test of the McClosky limestone from 3276 to 3282 ft, flowed gas in 4 min and recovered 550 ft of dry oil. C. A. F.

295. Humble well marks new area. Anon. *Petrol. Engr*, Nov. 1950, **22** (12), B-88.—The first well in the Timbalier Bay–Block 52 area, Terrebonne Parish, Louisiana, on a 4-hr test flowed 505 b.d. from 12,111 to 12,114 ft. Tubing pressure was 2600 p.s.i. C. A. F.

296. Shell finds new zone in south Louisiana. Anon. *Petrol. Engr*, Oct. 1950, **22** (11), B-96.—3 Continental Land and Fur Co., Terrebonne Parish, Louisiana, on completion flowed 325 b.d. and 1,506,000 cu. ft. gas/day from 6514 to 6534 ft. Total depth was 12,758 ft. G.O.R. was 4356 : 1. C. A. F.

297. Deep zone opened. Anon. *Petrol. Engr*, Nov. 1950, **22** (12), B-84.—1 Noah P. Harrington *et al*, Vermilion Parish, Louisiana, on test flowed 59 b.d. condensate and 3,000,000 cu. ft. gas/day from 11,543 to 11,552 ft. C. A. F.

298. Finds new producing zone. Anon. *Petrol. Engr*, Oct. 1950, **22** (11), B-96.—A new producing zone has been found in the Lottie field, Point Coupee Parish, Louisiana. The discovery well flowed 528/591 b.d. of 51·9° oil from 10,220 to 10,230 ft. C. A. F.

299. Second well discovered in Gulf of Mexico. Anon. *Petrol. Engr*, Nov. 1950, **22** (12), B-88.—A-3, 25 miles offshore from St Mary's Parish, Louisiana, had an indicated potential of 448 b.d. of 36·6° oil from the 3 Miocene sand at 11,372–11,392 ft. G.O.R. was 860 : 1. C. A. F.

300. Carter finds new oil area. Anon. *Petrol. Engr*, Oct. 1950, **22** (11), B-99.—1 Clyde Graham, 13 miles south of Feriday, Concordia Parish, Louisiana, on completion had an estimated potential of 300 b.d. of 44° oil from the Wilcox at 5976–5982 ft. Nearest Wilcox production is at Glen Aubin, 8 miles to the southeast. C. A. F.

301. Records broken by independent wildcat. E. Sterrett. *World Oil*, Oct. 1950, **131** (5), 107.—Tung Oil Corporation 1, 2 miles from Wiggins, Stone County, Mississippi, has reached 18,370 ft. Coring has been continuous below 14,450 ft. The well was spudded in 1946, but drilling operations have not been continuous.

The derrick is 136 ft high, and there are three oil-fired field " pots " and two mud-pumps. Wire-line is 1⅛ inch dia. Oil-based mud is used, weighing 8·8 lb/gal.

The well has not shown any potential production, but is providing invaluable

geological data. Bottom-hole temp is about 320° F. A table is included showing depths of marker horizons. The Smackover was topped at about 17,100 ft.

C. A. F.

302. Stratigraphy of Lower Mississippian rocks in southwestern Missouri. C. P. Kaiser. *Bull. Amer. Ass. Petrol. Geol.*, **34** (11), 2133–75.—General confusion, based largely on lack of specific detailed information, has existed for some time with regard to Mississippian stratigraphy in southwestern Missouri. Such reference as has been made to the geology of Mississippian rocks in this part of Missouri of recent years has been largely of limited scope and with reference to small areas. Accordingly, it seemed logical to the author to attempt a comprehensive examination of the entire area.

An attempt was made to examine as many outcrops as possible of Mississippian rocks in southwestern Missouri, and sixty-four locations were chosen for detailed study. Insoluble residues to aid in lithologic determinations were made in the laboratory from 176 hand samples, most of which were from Kinderhookian rocks. In addition to studying surface exposures, most of the cores from core holes drilled by the U.S. Army Engineers for the proposed dam near Osceola, St Clair County, were examined; and samples and drillers' logs from several wells in western St Clair County and eastern Vernon County were studied.

The basal Mississippian Sylamore sandstone is thin, locally developed, and poorly exposed in southwestern Missouri. The Chattanooga shale occurs only in the extreme southern part of southwestern Missouri, and there are no post-Chattanooga pre-Chouteau rocks. The Chouteau, Northview, and Sedalia formations are present in the northern part of the area studied, but thin southward and are not present along the southern border of the state.

It is recommended that the term Compton be dropped as a synonym of Chouteau. The Sedalia formation, as defined by Moore, is recognized as a valid formation, but it is Kinderhookian rather than Osagian in age. The lower two-thirds of the Sedalia formation is a facies of the Northview formation. The upper non-cherty part of the Sedalia is much more extensive than the underlying part and overlaps the Northview formation over a wide area.

Evidence is presented to prove that a part of the beds that have been referred to the St Louis formation in western Dade County should be referred to the Keokuk.

A number of very complete stratigraphic sections are presented. E. N. T.

303. Status of Dry Creek shale of Central Montana. C. Lochman. *Bull. Amer. Ass. Petrol. Geol.*, **34** (11), 2200–22.—The first published description of a Cambrian section in Montana was of the section along the north bank of the Gallatin River about 5 miles above its mouth.

The name " Dry Creek shale " was originally proposed by A. C. Peale in 1893 as a member of the Cambrian Gallatin formation. In 1899 Weed used the same name for an Upper Cambrian formation which he defined from the Little Belt Mountains of Central Montana, and subsequently the name has been used for a supposed Upper Cambrian formation at or near the top of the Upper Cambrian sequence of Central Montana. Recently Sloss and Laird (1947) demonstrated conclusively that most of the lithic units of Weed's Dry Creek shale formation belong to the Devonian cycle of sedimentation.

In August 1949 the author re-examined the key sections in the Horseshoe Hills between Logan and Dry Creek, Montana, located in each section Peale's Dry Creek shale unit, and determined its position with reference to the now recognized base of the Devonian and the Upper Cambrian faunal zones present in each section. The history of the usage of the name is here summarized, and the salient features of the Horseshoe Hills sections are discussed, with explanations about the numerous discrepancies.

E. N. T.

304. Nebraska has big gas well. Anon. *Petrol. Engr*, Oct. 1950, **22** (11), B-100.—Bosley 2, in the Big Springs area, Deuel County, Nebraska, flowed approx 10,000,000 cu. ft. gas/day from the First Sand, topped at 3268 ft. C. A. F.

305. New Mexico wildcat struck. Anon. *Petrol. Engr*, Nov. 1950, **22** (12), B-90.—1-A Brownfield, 15 miles north of the Denton field, New Mexico, flowed 416 brl oil in 18 hr from 11,785 to 11,840 ft. Devonian was topped at 11,790 ft. C. A. F.

306. Big well for Ohio. Anon. *Petrol. Engr*, Oct. 1950, **22** (11), B-100.—6 S. H. Cooperrider, Licking County, Ohio, was completed for 625 b.d. from the Clinton sand from 3007 ft. C. A. F.

307. Geology of Eola area, Garvin County, Oklahoma. R. M. Swesnik and T. H. Green. *Bull. Amer. Ass. Petrol. Geol.*, **34** (11), 2178–99.—The Eola field, in southern Garvin County just north of the exposed Arbuckle Mountains, is an extremely interesting pool from several viewpoints. Its complex structure, great productivity, and exceptionally difficult and costly drilling are all factors, but by far the most important aspect is that a proper interpretation of very excellent subsurface data in dealing with the complex structure may further help to decipher the geologic history of the Arbuckle Mountains.

The Eola field is on the subsurface extensions of these mountains, and is remarkable in the structural complexities revealed by the drill. Ten wells have been drilled, of which seven have been producers. These tests indicate the major structural feature of the area to be a west–northwest striking fault, with the magnitude of the stratigraphic displacement measurable in miles. Highly folded and faulted pre-Deese strata north of the fault form an overturned syncline whose axial plane dips southward. Oil and gas are controlled by local closure along secondary faults on the north and normal flank of the syncline.

The interpretation of these data indicates two major periods of diastrophism: post-Springer—pre-Deese and post Hoxbar—pre-Pontotoc. Evidence concerning the relative importance and intensities of these orogenies is discussed. Cross-sections are presented as an aid to deciphering the geological structure and history.

Oil is produced from the basal Bromide sand of Ordovician age at depths of 10,000–11,000 ft. The accumulative pipeline runs from January 1947 to April 1950, total 941,609 brl, the discovery well contributing 424,402 brl of this amount.

E. N. T.

308. Sinclair well flows 509 brl of oil an hour. Anon. *Petrol. Engr*, Nov. 1950, **22** (12), B-84.—1 Moore-Mullican Unit, Garvin County, Oklahoma, on test flowed 509 b.d. of 41° oil from the first Gibson sand at 6980–7007 ft. Gas production was 575,000 cu. ft/day. C. A. F.

309. Tulsa County, Oklahoma, has new oil pool. Anon. *Petrol. Engr*, Oct. 1950, **22** (11), B-96.—1 Katy Right of Way, Tulsa County, Oklahoma, had an estimated potential of 70–80 b.d. oil and 2,500,000 cu. ft. gas/day from the Burgess sand, topped at 1380 ft. C. A. F.

310. Gulf Coast has new pool. Anon. *Petrol. Engr*, Nov. 1950, **22** (12), B-88.—1 Sharp *et al*, Brazoria County, Texas, 5 miles southeast of Arcola, reported 175 b.d. from 7609 ft. C. A. F.

311. Discover Wilcox production. Anon. *Petrol. Engr*, Nov. 1950, **22** (12), B-88.—1 Pearle Wooten, Colorado County, Texas, produced 1,400,000 cu. ft. gas/day and 33 brl of 52° distillate per 1,000,000 cu. ft. gas from 8512 to 8540 ft. The well is approx 5 miles southeast of the Columbus field. C. A. F.

312. Sunray reports discovery. Anon. *Petrol. Engr*, Nov. 1950, **22** (12), B-90.—J. C. Dillworth 1, Live Oak County, Texas, on test flowed 2600 M.c.f. gas and 64 b.d. of distillate from 7173 to 7214 ft. 128 ft of Wilcox pay were found. C. A. F.

313. Find new zone, Milroy pool. Anon. *Petrol. Engr*, Oct. 1950, **22** (11), B-99.—2 Harley, in the Milroy pool, Stephens County, Texas, flowed 322 brl in a 12-hr test of a new Pennsylvanian pay, topped at 1770 ft. C. A. F.

314. Shallow pay in Scurry reef area. C. J. Deegan. *Oil Gas J.*, 27.4.50, **48** (51), 221.—J. A. Chapman Co. 6 Cogdell in southern Kent County, West Texas, has flowed 1232 b.d. through a ¾-inch choke from a pay nearly 2000 ft above the normal Canyon reef. The pay is at 4910 ft, and is either Wolfcamp or Cisco. G. D. H.

315. New Texas oil pool proved. Anon. *Petrol. Engr*, Nov. 1950, **22** (12), B-90.—2 N. W. Willard, southern Terry County, Texas, after acidizing flowed 912 b.d. of 43° oil from 8500 to 8560 ft. Pay is basal Permian limestone. C. A. F.

316. Union of California finds 92-ft oil sand, Texas. Anon. *Petrol. Engr*, Oct. 1950, **22** (11), B-99.—1 Laura, 4 miles southeast of Brownfield, Terry County, West Texas, found a 92-ft pay section and on test between 9910 and 9920 ft flowed about 10–15 brl/hr of good oil. In a test of the interval, 9991–10,002 ft, the well flowed at 82 brl/hr. C. A. F.

317. Early Devonian gas in northern West Virginia and pre-Devonian oil prospects. F. Reeves and P. H. Price. *Bull. Amer. Ass. Petrol. Geol.*, **34** (11), 2095–132.—During the past twenty years gas sands in the lower part of the Devonian have been the objective of most exploratory drilling in West Virginia and southwestern Pennsylvania.

Since the discovery of gas in the Oriskany sand in north-central Pennsylvania in 1930, nineteen deep wells have been drilled to the Oriskany on seven major folds 30–40 miles southeast of the old oil and gas fields in northern West Virginia. This exploratory drilling has resulted in the discovery of two gas fields, known as the Tarra Alta and Canaan Valley fields, situated respectively 25 and 50 miles south of the Summit field in Fayette County, Pennsylvania. The Tarra Alta field has eight gas wells, and the Canaan Valley field three gas wells. The extent of neither field has been fully defined.

Gas is produced from the Huntersville chert and the underlying Oriskany sand, which are encountered at depths ranging from 4900 to 8250 ft. Yields vary from ½ million to 4 million cu. ft/day/well. Small flows of gas also have been obtained in the Benson and Speechley sands and Helderberg limestone in a few wells. Shows of oil have not been reported.

The authors suggest that no factual or theoretical data can be presented to disprove the possibility that oil may be present in pre-Devonian formations in some of the major folds of the region, where fracturing and solution weathering may have developed secondary porosity. It is possible that the hydrocarbon content of the entire pre-Trenton Paleozoic section may have migrated along fissures to the Trenton and be held there by the impermeable cover of Upper Ordovician shale.

A comprehensive stratigraphic summary is presented, and the gas and oil prospects are examined individually. It is suggested that the logical place for a test of the older Paleozoic formations is on a major fold where fracturing and solution weathering may be expected and where erosion has exposed the formations at sufficient depths so that the Ordovician and Cambrian objectives are within reach of the drill and yet have a good cover. A 10,000-ft well starting near the base of the Devonian should penetrate most of the Paleozoic unless thrust faults of considerable displacement are encountered, and, of course, this is a contingency that must be taken into consideration in planning deep tests. It is possible that the hydrocarbon content of the entire pre-Trenton Paleozoic section, in a fractured major fold, may be segregated within the Trenton and retained there by the cover of Upper Ordovician shale. Because of this possibility, it may be advisable to regard the Trenton as the principal objective. E. N. T.

318. Wyoming discovery seen. Anon. *Petrol. Engr*, Nov. 1950, **22** (12), B-88.—1 Anderson, Cheyenne County, Wyoming, 12 miles southwest of the Huntsman field, on test flowed 15,000,000 cu. ft. gas/day from the D sand. Total depth was 5300 ft. C. A. F.

319. Well tests 52 bbl an hour. Anon. *Petrol. Engr*, Nov. 1950, **22** (12), B-88.—Government 84–32, Slick Creek, Washakie County, Wyoming, on test produced 52 brl/hr of 31·7° oil from Embar phosphoria at 10,464 to 10,544 ft. 800,000 cu. ft. gas were recovered in 24 hr. C. A. F.

320. Wyoming discovery made. Anon. *Petrol. Engr*, Nov. 1950, **22** (12), B-90.—A well drilled 2 miles east of Worland in Wyoming flowed 1200 b.d. of 32° oil. The well was bottomed at 10,554 ft in Embar limestone. C. A. F.

321. Venezuelan well drilled. Anon. *Petrol. Engr*, Nov. 1950, **22** (12), B-86.—A well on the western edge of the Ruiz field, eastern Venezuela, on tests of the Chaguaramas at about 2964 ft, flowed 414 b.d. of 38·9° oil. C. A. F.

322. Complete Edmonton well. Anon. *Petrol. Engr*, Oct. 1950, **22** (11), B-94.—Acheson 1, 8 miles west of Edmonton, on test had estimated potentials up to 1500 b.d. 121 ft of D_3 reef were discovered above oil–water level. C. A. F.

323. Socony gets Canadian well. Anon. *Petrol. Engr*, Oct. 1950, **22** (11), B-94.—1 Flint, 50 miles southeast of Edmonton, Alberta, found 29 ft of D_3 limestone at 4507 ft. On tests the well flowed 700, 200, and 575 b.d. of 33·8° oil. G.O.R. was 500 : 1. C. A. F.

324. Alberta strike made. Anon. *Petrol. Engr*, Oct. 1950, **22** (11), B-99.—1 Big Valley, about 18 miles south of the Stettler field, on test had an estimated flow of 1,500,000 cu. ft. gas/day from 5237 to 5252 ft. Oil flowed at 100 brl/hr. Pay is D_3. C. A. F.

325. Leduc oil field, Alberta, Canada. T. A. Link. *Bull. Amer. geol. Soc.*, Mar. 1949, **60** (3), 381.—The Leduc field was discovered in February 1947. Production was found in two Devonian limestone–dolomite zones, the D_3 and D_2.

The D_3, the main pay, is 0–560 ft thick. There is considerable fracturing, and porosity and permeability are locally high. Pyrobitumen has been found in the limestone in the gas cap.

The D_2, which varies from 140 to 160 ft, is a dense granular dolomite with intergranular and cavernous porosity. Cementation has locally closed the pores of the dolomite. D_3 and D_2 oil is believed to be Devonian and indigenous to the reservoir.

A structure map on the Lower Cretaceous shows a north-east–southwest-trending nose which may be closed on the northeast. Although there is structural relief, hydrocarbon accumulation in the Devonian is primarily controlled by reef development.

The shallower pay (D_3) is a " regressive " reef and the D_2 " transgressive." The theoretical development of these types of reefs is described in detail. Both types could produce blanket or compound reefs with considerable lateral extension.

Regressive reefs are characterized by four depositional facies : (1) back reef evaporites and clastics; (2) massive reef accumulations; (3) fore-reefs with well-bedded clastics; (4) basin sediments of shales and limestones. The transgressive reef has no back reef facies. Hybrid reefs could develop with pulsating transgression and regression. Diagrams are included to show reef development during transgression and regression, and the hypothetical development of the D_3 reef.

Maps show the oil and gas fields of Alberta, the tectonic features of Western Canada, and the structure of the Leduc field.

Fourteen references are included, and there are nine photographs of limestone cores. C. A. F.

326. Navy finds two wells on Alaskan coast. Anon. *Petrol. Engr*, Nov. 1950, **22** (12), B-90.—Two minor oilfields have been discovered in Alaska. A well at Umiat is producing 500 b.d. of high-gravity oil, and three more shallow wells are planned in the area. Productive capacity of the field at Cape Simpson is unknown. C. A. F.

327. Production of natural gas in France. J. Allison. *Petrol. Times*, 22.9.50, **54**, 647.—Oil seeps in France have been known for some years, chiefly in the Ardour basin and in the Herault area in southern France. 150 wells, totalling 90,000 ft in depth, were drilled after the first world war, resulting in the discovery of the Gabian oilfield. 22,000 tons had been extracted from the field by 1935, when it became exhausted. Gas has since been found at St Marcet in the foot-hills of the Pyrenees. The discovery well on test made over 6,000,000 cu. ft/day.

Exploration has subsequently been centred in this area, and the St Marcet field is under development. Average depth of wells drilled in the area in 1947 and 1948 was 9184 ft. A well was drilled to 13,623 ft in 1947. The field produces oil and gas. Gas production, which is dominant, comes mainly from Middle Cretaceous dolomite and breccia at about 5000 ft beneath Senonian shales. The structure is an anticline

with a core of Trias. Oil is present in Jurassic dolomite on the south flank of the structure. Crude is of high gravity with 22% gasoline, 6% kerosine, 11% gas oil, and 48% lub oil. There is no sulphur. Gas production is expected to exceed 35 million cu. ft/day by 1952. Cumulative gas production to the end of 1948 was 24,500 million cu. ft.

A pipeline system has been built to distribute the gas. A gasoline plant of 42 million cu. ft/day capacity has been built at Boussens. Gas is supplied to Toulouse, Bordeaux, and elsewhere. C. A. F.

328. Oil the elusive—the search for oil in the Australian region. A. Wade. *Petrol. Times*, 1.12.50, **54**, 806.—Oil shows have been found in widely separated areas in Australia, Papua, and in the Mandated Territories of New Guinea. Petroliferous rocks range from very old to very young.

Oil search in New Guinea began before the first world war, and between 1913 and 1920 there was some production from wells at Upoia on the Vailala River. Liberalization of petroleum legislation in Australia in the early thirties was followed by exploratory work in Queensland, the West Kimberly district, and elsewhere. There has been some success in New Guinea, where a few fields have been found. C. A. F.

Geophysics and Geochemical Prospecting

329. Exploration speeded in Peace River by aeromagnetic survey. T. O'Malley. *Petrol. Engr*, Oct. 1950, **22** (11), B-36.—An airborne magnetometer survey of 16 million acres has been completed in the Peace River area, Alberta. Cost of the survey was about 150,000 dollars. The instrument used was the Gulf high-sensitivity airborne magnetometer, which has been used successfully in the Bahamas and elsewhere. Approx 800,000 acres were covered per day at an altitude of 1000 ft. Parallel traverses were flown 1½ miles apart, and a continuous magnetic record was obtained, accurate to 1 or 2 γ. A duplicate ground-based magnetometer was used for detecting magnetic storms. Maps contoured at 5–10 γ are to be compiled. C. A. F.

Drilling

330. Integrated submersible drilling barge. E. Sterrett. *World Oil*, Apr. 1949, **128** (13), 120.—A detailed description is given of the Gulf Coast Drilling Company Barge No 1, designed, built, and equipped by one concern, ensuring, thereby, the min of improvisation. The barge, of 140 × 52 × 11 ft, was designed to accommodate drawworks of rated input power of 1400 h.p. for drilling to a depth of 15,000 ft with 4½ inch O.D. drill pipe. The height of the derrick is 136 ft.

The principal feature is a compact design which permits also the flexible and safe operation of the equipment. A. J. H.

331. Controlled vertical drilling. W. C. Main. *World Oil*, Apr. 1949, **128** (13), 95.—The definition and the necessity of vertical and straight holes are both governed to a great extent by local factors, such as property lines, well spacing, and formations. In the general case, however, controlled drilling is of importance for the maintenance of sound equipment.

Straight holes are defined in this paper as those which do not change in the vertical angle by more than 6 min/100 ft, and in the lateral angle by more than 3°/100 ft; vertical holes are those which do not deviate, at any point, by more than 2° from the vertical and 75 ft from the vertical centre line of the well.

In directional drilling the deviation is regular, while for controlled drilling the angle correction may be a reversal. Thus the principal danger lies in an excessively rapid reversal. Deviation is of greater danger near the top of the hole; thus to avoid keyseating in a well of 10,000 ft depth the change in the angle should not exceed :—

10 min/100 ft in the upper half;
15 min/100 ft in the lower half.

Although under pure bending a pipe may safely withstand a bend of 3½°, the combined stresses due to bending, torque, and compressive stress waves require that bending stresses be minimized. A. J. H.

332. Directional drilling in off-shore operations. G. B. Parks, *World Oil*, June 1949, **129** (2), 95.—A review of some of the problems encountered in the drilling of two high-angle deviation wells in the Gulf Coast offshore drilling operations.

Plans and vertical projections and drilling data of the wells are given, and the procedure discussed in detail for the first directional well on that lease. A. J. H.

333. Combination casing strings. A. C. Temmel. *World Oil*, June 1949, **129** (2), 111.—Max efficiency and safety are obtained only by the careful selection of sizes. The design problems associated with these strings are considered in detail.

Tables and graphs are given. A. J. H.

334. A multi-thermometer method for temperature measurement in bore-holes. S. C. Mossop. *J. Chem. metall. Soc. S. Africa*, Sept. 1950, **51** (3), 120–2.—A modification of Krige's method for measuring temp in deep, narrow boreholes is described. The technique was developed while attempting to calculate the rate of heat flow from the earth's interior.

Several thermometer containers were attached to the wireline to give temperatures at various depths in a single run. Max thermometers in two ranges were used, 15–45° C and 30–60° C, graduated to 0·1° C. Each thermometer was enclosed in a close-fitting metal tube. Three of these tubes were enclosed in an outer sheath, to which was attached the suspension wire, which was 0·036-inch dia piano wire. Ten sheaths were used, attached to the line at 200-ft intervals. Under test, the thermometers reached borehole temperature within 15 min. Temp readings at 200-ft intervals, from 3000 to 4800 ft, were obtained in 4 hr.

Possible sources of error are briefly discussed.

Drawings of the thermometer tubes and containers are included, and seven references given. C. A. F.

335. Compressibility of bulk agents to combat lost circulation. J. I. Morris. *World Oil*, Apr. 1949, **128** (13), 104.—The characteristics, as estimated graphically, of the seven types of agents under test enabled them to be placed in two main groups, one being of the more fibrous materials.

The paper deals in detail with the experimental procedure, the methods of calculation, the compressibility factors, and their application to field use. A. J. H.

336. Transporting rotary bit cuttings. G. C. MacDonald. *World Oil*, Apr. 1949, **128** (13), 114.—The efficiency of the removal of cuttings is dependent on the upward mud velocity and the downward cutting velocity. Thus the discharge at the surface of the small flat particles may indicate that the larger round particles are not being removed. Adjustment of the pumping system is then necessary. Although higher annular velocities are obtained by the use of larger drill pipe, their use is limited by the necessity of safe clearance for possible fishing operations.

The use of graphs, seven of which are reproduced, greatly simplifies the estimation of suitable running conditions, but in every case allowance must be made for irregularities in the hole. A. J. H.

337. Mud flow through oil field diamond core bits. B. J. Westman. *Petrol. Tech.*, July 1950, **2** (7), 8–9.—Early diamond bits invariably failed along the crown. This could not be attributed to junk damage. In an attempt to remedy this more diamonds were put in the crown, but this, while reducing shock per diamond point, increased mud velocity by reducing clearance. High mud velocity eroded the matrix. One company, however, which used 6¼-inch × 3½-inch insert-type core bits, gradually reduced the number of inserts from twenty with 250 carats to eight with about 100 carats. These bits gave greater speed of penetration, lower footage costs, and greater bit economy. The principal wear was damage to outside gauge diamonds due to accumulated trip-feet. Mud erosion was nil, and bit blanks were reset a number of times, thus saving expense.

Subsequently a "port bit" was developed with tapered holes from each channel just outside the crown centre into the bit taper just above the major restriction at the inside gauge. This gave higher circulation capacity, eliminating bit bounce, prolonging bit life, and increasing penetration rate. Fluid velocities less than 2500 ft/min

across the bit crown are desirable. 10–20 min/ft in shale is often common with such a bit due to prevention of balling. Junk damage can be reduced by thoroughly cleaning the hole before coring, and a multiple-pole magnetic junk retriever plug is being developed to pick up junk which cannot be circulated out past the bit. High-clearance bits minimize diamond wear along the crown and inside gauge; it is also possible to wash out core fragments which formerly packed in the inner taper above the bit crown. This wedging action tends to break up the core, and can lead to core jamming. G. D. H.

338. Chemical treatment of cement-contaminated drilling muds. R. W. Jones. *World Oil*, May 1949, **129** (1), 106.—It was observed that the effects of cement-contamination on muds were much reduced when these had been subjected to sodium acid pyrophosphate treatments. Investigations were then carried out on the possible pretreatment of muds by the addition of mono-sodium *ortho*-phosphate; this compound was found to restrict changes in visc, water loss, and pH.

Experimental procedures and relevant theory are discussed, and comparative graphs and references given. A. J. H.

339. Controlling anhydrite contamination in Rocky Mountain drilling muds. D. D. Varnell and W. B. Kimbrell. *World Oil*, June 1949, **129** (2), 129.—Contamination of drilling muds has resulted in decreased visc and increased fluid loss.

Several treatments are used, including the use of barium carbonate and of soda ash. The former causes a precipitation, resulting in the removal of the contaminant and the treater, as barium sulphate and calcium carbonate. Each treatment has to be considered on the merits of the local conditions. A. J. H.

340. Lime content of drilling mud—calculation method. M. D. Nelson and T. E. Watkins. *Petrol. Tech.*, July 1950, **2** (7), 10.—A method of mud analysis for lime content involves titrating 1 ml of mud and 1 ml of filtrate each with 0·02 N-H_2SO_4 to the end-point with phenolphthalein. If P_m is the volume for the filtrate the lb lime/brl of mud was given as $(P_m - 1{\cdot}3P_f) - 0{\cdot}6$. The formula has given erroneous results for muds either high or low in solids, and a preferable relationship is 0·26 $(P_m - F_1P_f)$. F_1 is the volume fraction of liquid in the mud. This suggested procedure ignores lime dissolved in the mud.

A nomogram and several examples are given. G. D. H.

341. Control of heaving shale with sodium silicate muds. W. V. Vietti. *World Oil*, June 1949, **129** (2), 104.—The difficulties in drilling through strata of heaving shale have in many cases caused the abandonment of certain wells. Originally, purely mechanical solutions to these problems were attempted, but research into the use of silicate mud has shown that this is an economic proposition.

Heaving shales cause the drill pipe, or casing-string, to seize, as a result of lateral expansion of the shales and the caving of the well wall. Preventive measures of restricting the swelling of the shale have permitted the drilling through of these regions.

The estimation of the depth of heaving shales is possible in proven areas, and current practice is to case the well to that depth before using silicate muds. In unknown areas, however, tests should be made on cores; in these tests it is essential to select a fresh core, as the drying effect on an exposed core is such as to give inconsistent results.

Of particular interest is the greater initial production obtained from wells drilled with silicate muds. This is due to the reduction of the swelling of clay particles in the sand itself.

Practical details are mentioned and references given. A. J. H.

342. The S.P. log in shaly sands. H. G. Doll. *Petrol. Tech.*, July 1950, **2** (7), 205–14.—A theoretical study of the S.P. log is presented. Opposite a thick clean sand the S.P. values reach a limiting figure which is described as the "static S.P." of the clean sand. This depends on the salinity of the connate water relative to the mud, and on the pressure differential, but not on the sand resistivity. The limiting value of the S.P. opposite a thick, shaly sand is smaller, and is known as the "pseudo-static S.P." of the shaly sand. This is a function of the amount and resistivity of the shale, and other resistivities. The detailed action of the local S.P. currents in the shaly sands is

discussed. It is concluded that the pseudo-static S.P. of a shaly sand is smaller if the sand is oil-bearing than if it is water-bearing. Usually the amount of shale is less in the centre than in the top and bottom of a sand.

There is a discussion of the computation of the resistivity of the connate water from the pseudo-static S.P. of a shaly sand, of the effect of the use of two successive muds of different salinities, and of the effect of electro-filtration e.m.f.'s. G. D. H.

343. Applications of surface plugging agents in oil and gas wells. A. A. Townsend. *World Oil*, Apr. 1949, **128** (13), 124.—The effectiveness of plugging agents is dependent on the S.G. and particle size of the fillers; these are divisible in two groups, insoluble and acid soluble.

The principles of operation are described for the varying requirements, such as reduction of permeability or complete shut-off. The selective plugging of formations permits the acidizing of those zones for which the treatment is intended. The use of fillers for temporarily closing off, or blanketing, the bottom of the well is also described. A. J. H.

344. A well completion technique for North Louisiana. J. M. Reynolds. *World Oil*, May 1949, **129** (1), 102.—Of special application in low-pressure areas, this technique involves the use of both rotary and cable-tool drilling. The first part of the well is drilled with a medium-depth rotary rig and casing run to a depth just above the producing zone. This is then penetrated by the conventional cable tool, thereby allowing the drilling to proceed under a low hydrostatic head, reducing formation contamination.

The development of a combination sand pump and drilling bit has greatly improved the method of cable-tool drilling. The chippings are drawn into the bailer by the suction effect of the plunger, which rises when the unit is pulled upwards from the surface. The length of plunger stroke determines when the bailer is full.

A diagrammatic sketch is given of the combination tool, and the drilling procedures adopted on various leases are explained. A. J. H.

Production

345. The application of radioactive tracer techniques to the study of the movement of oil in sands. S. E. Coomber and E. N. Tiratsoo. *J. Inst. Petrol.*, 1950, **36**, 543–58.—The results are described of preliminary investigations into the possibility of using radioisotopes in following the movement of oil in sand bodies. The basic theory of radioactive tracer work is outlined, and details are given of the apparatus employed and the technique recommended. Methods of measuring the absorption properties of laboratory materials for the radiation of radioactive iodine are described, and the bearing of the results on quantitative measurement is discussed. Experiments have been made on the movement of oil through sands of differing grades and its accumulation at a coarse–fine interface in order to define the technique and reliability of such measurements in dynamic conditions. The potentialities of these techniques in reservoir-engineering investigations are outlined. A. R. W. B.

346. Helium tracer gas studies in the Cabin Creek, W. Va., oil and gas field. E. M. Frost, Jr. *U.S. Bur. Mines Rep Invest. No.* 4715, Aug. 1950.—Helium, injected with natural gas in petroleum reservoirs, is particularly useful as a tracer gas for obtaining data with respect to secondary recovery, recycling, pressure maintenance, and other purposes. Its properties and functions in such work are briefly described. This report covers three tests, using 1 and 5% helium in natural gas and essentially pure helium, carried out by the Bur. Mines and the Pure Oil Co. in three test injection wells at the Cabin Creek field, Boone and Kanawha Counties, W. Virginia. Charts show the injection wells and the many producing wells from which the tests results were obtained. The data are discussed with respect to: time–distance relationships, determining concentration of injected gas in the producing wells, reservoir pore-vol and volumetric calculations, ratio of injected gas to gas produced, and vol of gas-flow channels. W. H. C.

347. Oil production by gas and flooding. P. J. Jones. *World Oil*, Apr. 1949, **128**, (13), 176; May 1949, **129** (1), 166; June 1949, **129** (2), 170.—Parts 6–8 of a

series covering a radial reservoir; declining reservoir pressure; and displacement of oil by flooding. A. J. H.

348. North Cowden field—gas injection project. M. H. Rahmes. *World Oil*, Oct. 1950, **131** (5), 153.—The gas-injection project in the North Cowden field, Texas, is one of the largest projects of this type in the U.S.A. It involves a pressure-maintenance programme planned to increase ultimate recovery from the Graysburg formation. All the gas produced from the field is to be processed.

Productive area of the field is about 27,000 acres, on which 680 producers have been drilled on 40-acre spacing. Cumulative production to the end of 1949 was approx 79 million brl. The structure is an elongated anticline with about 800 ft relief. Pay is Permian Graysburg sand and dolomite, topped at approx 4100 ft at the crest of the structure. Dissolved-gas drive formerly operated, and there was no effective water drive. Original b.h.p. was approx 1800 p.s.i., declining to 727 p.s.i. at the end of 1949.

In the injection project, the field has been divided into blocks of about 640 acres. One injection well is being drilled in each block, and a min of two in each is planned. A high-pressure gasoline and injection plant is operating at a capacity of 50 million cu. ft/day. The compressor plant has ten 1600-h.p. units in two houses. Gas injection commenced in May 1950. C. A. F.

349. Flooding with re-used water. F. Squires. *World Oil*, Apr. 1949, **128** (13), 152.—This process consists in flooding with water from a sand above the producing sand, and has the advantage of reducing the costs of water supply.

The well pattern is the conventional five-spot. With sands of low permeability, an additional source of pressure energy may be required. In this case air is pumped down the annulus of the outer wells, thereby forcing water down the central well into the lower sand; oil is then produced up the tubing of the outer wells. A. J. H.

350. Acidizing techniques in sand formations. E. N. Jones. *World Oil*, Apr. 1949, **128** (13), 168; May 1949, **129** (1), 156.—The primary purpose of acidizing is to restore the sand body to its original condition; thus the use of muds having good wall-building properties in the initial drilling reduces the necessity of acidizing.

Techniques in acidizing are numerous and must be developed for each individual well, consideration being given to its previous records. Subsequent to treatment it is essential to remove the acids and loose particles. Components of "mud acids" include hydrochloric and fluoric acids, corrosion inhibitors, and detergents.

Experimental evidence confirms that mud acids do not appreciably affect the cement of the well, although a thin coating was observed on some test specimens of cement. Cement removed from wells is attributed to contaminated cement and not to the direct action of mud acids.

The importance of acidizing in conjunction with squeeze-cementing operations is discussed. Specific examples are taken.

Data given in tabular and graphical forms. A. J. H.

351. Gas dehydration at the well head. L. L. Laurence and L. S. Reid. *World Oil*, June 1949, **129** (2), 156.—The process described obviates the need for extensive protection at the gathering centre. The standard well-head unit may have a through-put capacity in the region of 1 million cu. ft/day.

Operational data are given. A. J. H.

352. Mechanical removal of free water from high-pressure gas lines to prevent formation of hydrates. A. F. Barry. *World Oil*, May 1949, **129** (1), 137.—The formation of gas hydrates presents problems which increase in conditions of high pressure and of low surface temp. Thus in the Condensate field at Lake Creek, Montgomery County, Texas, formation of hydrates under 1800 p.s.i. pressure caused the shut-down for 20% of the winter operating time; hydrates began to form at 70° F.

The development of the water knock-out device resulted in the reduction of water formation from 6300 lb daily to 103 lbs daily. The unit, which is described diagrammatically, consisted of a baffle, and a means of "dumping" automatically.

Flow data obtained on the field are given. A. J. H.

353. Improvements in the X-ray saturation technique of studying fluid flow. F. Morgan, J. M. McDowell, and E. C. Doty. *Petrol. Tech.*, July 1950, **2** (7), *A.I.M.M.E. Tech. Paper No.* 2881, 183–94.—Improvements have been made in the X-ray method of measuring liquid saturation and saturation distribution in cores. Two identical D.C. amplifiers have been added to measure continuously the intensities of the X-ray-tube output and the beam which has passed through the test core. The ratio of these intensities is automatically recorded. The core is driven automatically and in synchronism with the recorder chart so as to give a continuous curve showing the liquid saturation along the core. Scanning speeds range ½–1½ in/min. The theory of the method is given. By determining the ratio of the two X-ray-beam intensities simultaneously, the measurements are essentially independent of small fluctuations in tube supply voltage or current.

The results of several fluid-displacement studies are given. For cores cut into sections and pressed together discontinuities were observed at the junctions, while the expected rise in saturation at the retaining disc in capillary-pressure experiments was demonstrated. G. D. H.

354. Core analysis problems in secondary recovery. R. Staples. *World Oil*, Aug. 1950, **131** (3), 156.—Ultimate recoverable reserves must be accurately determined, and for this purpose a thorough knowledge of sand conditions, acquired from representative cores, is necessary.

The chief problem in core analysis is sampling and interpreting the data, especially where there are vertical lithological changes. The use of the diamond core barrel has reduced sampling errors by the increased core recovery.

The porosity and permeability of core samples must be determined. Horizontal permeability is very important, especially in oil-recovery calculations and in determining injection rates in secondary recovery.

Determination of oil visc is essential in a secondary recovery project. Water injection has been used in reservoirs containing oil having a visc as high as 35 cp. Gas injection has been used against oil having a max visc of 20–25 cp.

In the determination of true oil saturation of cores, it is necessary to calculate accurately the amount of core flushing or to use oil or oil-based mud. C. A. F.

355. Pore size distribution of petroleum reservoir rocks. N. T. Burdine, L. S. Gourney, and P. P. Reichertz. *Petrol. Tech.*, July 1950, **2** (7), *A.I.M.M.E. Tech. Paper No.* 2893, 195–204.—The relationship between equivalent pore-entry radius in samples of petroleum reservoir rock and the associated pore volume has been investigated. Data have been obtained by the mercury capillary-pressure technique, using pressures up to 1500 p.s.i. A single run took about 4 hr. The theory of the method and the construction of the apparatus are described.

The results of a number of investigations are given. Repeatability is generally good, a technique for regenerating the specimens being described. The capillary pressure measurements apparently caused no change in the samples. At 1500 p.s.i. mercury filled 53–85% of the pore space. The effect of shape, surface area, and wt of the specimens on the observations was investigated. A greater volume of mercury entered the pores at lower pressures when the cores were fragmented.

Mercury capillary-pressure curves are compared with those obtained by the porous-diaphragm method using gas to displace water.

Theoretical equations are given for calculating permeability from pore-size distribution, and the results of applying these equations are compared with measured gas permeabilities. An effective capillary length factor appears in the equations, and the comparisons show that the square of this factor varies from 2·6 to 11·3. G. D. H.

356. Porosity—permeability check by electric log. R. C. Barber. *World Oil*, Aug. 1950, **131** (3), 165.—Successful secondary recovery by water flooding depends primarily on injecting flood water into the proper zones under the most efficient pressures to effect a continuous movement of the oil ahead of the flood. Determination of porosity and permeability of injection zones is therefore of great importance. In the Bradford field water-flood project, the porosity and permeability of zones have been studied from electric logs run on injection wells.

Wells are electrically logged under different hydrostatic heads, giving differential

resistivity curves which are correlated with permeability. The technique makes use of short lateral curves in the Schlumberger log. Electrode spacing is a few inches, and the amount of water in the hole is carefully controlled. The first resistivity curve is obtained immediately after the sand is covered with water, and the second is run 2–3 hr after the first, when the sand is covered with a head of water not exceeding the flooding pressure. Increasing the hydrostatic head above flooding pressure causes fluid invasion of the sand and a corresponding decrease in the salinity of the formation water. The corresponding resistivity increase, which is proportional to the amount of water flooded through the formation, is a direct function of the relative permeability of the formation to water.

Examples of resistivity and differential resistivity curves are included.

Nine references are given. C. A. F.

357. Factors influencing dynamometer card shape. K. N. Mills. *World Oil*, Apr. 1949, **128** (13), 145.—The effects of vibration in the rod string are shown in the comparison between the ideal dynamometer card of a pump operating under 100% volumetric efficiency and under static loads, and those of pumping systems operating at various speeds.

In order better to understand the interpretation of the practical card, an introduction is given to the theory of wave formations. The two pump strokes are considered separately, but, in practice, the first and second series of stress waves are not independent of each other. Thus, waves reflected from the first series may combine to cancel the effect, or to augment it; damping may, therefore, appreciably affect the card shape. This fact was developed, and the use of elastic media has, in some cases, resulted also in the reduction of the polished-rod load. A. J. H.

358. Use of filler and plastics in sand consolidation. W. S. Stovall. *World Oil*, June 1949, **129** (2), 152.—Difficulties have been encountered when producing from Miocene sands of 2000–6000 ft depth, when the unconsolidated nature of the sands has given rise to excessive abrasion and " sanding up " of the producer.

The original procedure of screening proved unsatisfactory, especially when produced water corroded the screens, necessitating costly clean-outs. The use of plastics was found to be effective, although more than one application was needed; the presence of water in no way affected the consolidation of the sand.

Two types of plastic were used: filler plastic and consolidating plastic. Shrinkage of the plastic permitted the retention of the essential porous character of the sand.

A brief account is given of the method of application. A. J. H.

359. Tubing joints for high pressure wells. H. S. Kelly and G. G. Hebard. *World Oil*, June 1949, **129** (2), 145.—A short review of the design changes and improvements made since 1924. In 1938 a sub-committee of the A.P.I. was set to investigate the deficiencies in tubing design, which resulted in numerous failures at that time. The eight-round thread (drill pipe) form was adopted in 1938, and the use of the ten-round thread on 1¼- and 1½-in upset tubing continued.

Difficulty had been experienced recently in making up the eight-round thread tapered joint sufficiently tightly to serve in high-pressure pools, and recent investigations began of this design, particular attention being paid to hoop-stress.

Some of the features required by field practice, and not found in these joints, are summarized, and comparison is made between the A.P.I. and other joints. The use of these special tubing joints, although restricted by cost, may in certain areas be justified. A. J. H.

Oilfield Development

360. Exploration continues high in June. Anon. *World Oil*, Aug. 1950, **131** (3), 90.—741 exploratory wells were completed in the U.S.A in June 1950, compared with 770 in the previous month. About 25% of the tests were producers. 3924 exploratory wells were completed in the first six months of 1950, 8·9% higher than in the same period of 1949. To the end of June 1950, 524 new pools had been found. Of these, 379 were new fields and 145 new pays in known fields.

Tables show the results of exploratory drilling in the U.S.A. in June 1950 and the first six months, 1950–49, by districts; and the new oil, distillate, and gas fields and new pays discovered in June 1950. C. A. F.

361. Oil and gas developments in Kentucky during 1949. C. D. Hunter, J. Browning, W. S. Browning, and W. Diamond. *Petrol. Tech.*, July 1949, **2** (7), 83–91.—In 1949 460 oil wells, 196 gas wells, and 459 dry holes were completed in Kentucky. The oil output was 8,806,230 brl. Twenty-seven Corniferous wells were drilled in the White Creek field, an extension of the Big Sinking Field. 186 wells were drilled in the Big Sandy gas field, 149 being producers with a total open flow of 101,950 M.c.f. 2642 of the 4649 Big Sandy field wells produce from the Devonian black shale. Only 4·8% are natural producers; 38·5% have no open flow before shooting; only 16·2% produce over 100 M.c.f/day before shooting. These shales provide over 70% of the ultimate gas recovered in Eastern Kentucky.

The Poole Consolidated field has been extended with pay zones in the Palestine and Hardinsburg sandstones, and good shows in the Waltersburg and Tar Springs.

Tables summarize the 1949 drilling results by counties, and also for the Big Sandy gas field; the monthly oil production is given by counties; brief data are given on discoveries and extensions; western Kentucky fields are listed with cumulative and daily production. Maps show the positions of the fields and the producing formations. G. D. H.

362. 1950 looms as promising year in Mississippi oil and gas development. Anon. *Oil Gas J.*, 22.6.50, **49** (7), 260.—In 1949 eight new fields and two new reservoirs in old fields were found in Mississippi. So far in 1950 there have been six successful wildcats. One found lower Tuscaloosa production at 10,300–10,320 ft near Natchez in Adams County. Three Wilcox completions have been made between the Sibley area and the Armstrong–Ellispie field. There are now fourteen oil or gas pools in Adams County. The West Lincoln field in Lincoln County was opened with lower Tuscaloosa production at 10,646–10,684 ft.

In the Pickens field salt was met at 12,219 ft, and the Smackover had numerous shows. This deep well was completed at 7728–7737 ft (probably Travis Peak).

In southern Mississippi four fields are given about 8,000,000 M.c.f/month. Gwinville has seventy-five gas-condensate wells. Baxterville has a gas-condensate reservoir at about 7700 ft, and gives low-gravity oil at 8700 ft from 129 wells.

In 1949 Mississippi produced 37,885,120 brl of oil and condensate from thirty-six fields. Gas production was 108,978,452 M.c.f. About 35,000,000 M.c.f. of gas was reinjected at Brookshaven and Cranfield. 349 wells were drilled for oil or gas, and 112 wildcats gave eight oil or gas fields. To the end of February 1950, Mississippi had produced 251,735,095 brl of oil, of which 113,461,276 brl had come from Tinsley. G. D. H.

363. Oil and gas developments in Missouri during 1949. F. C. Greene. *Petrol. Tech.*, July 1950, **2** (7), 92.—Four oil, six gas, and twenty-one dry holes were completed in Missouri in 1949. The oil output was 32,027 brl from about ninety-nine pumpers; 23,340 M.c.f. of gas was sold from eleven wells. G. D. H.

364. Oil and gas developments in Nebraska during 1949. E. C. Reed. *Petrol. Tech.*, July 1950, **2** (7), 93–4.—Oil was found in the Gurley field of Cheyenne County. It is in the top of the Dakota at about 4400 ft. On test the flow was 17 brl/hr. There is a E.N.E.–W.S.W. faulted dome which was located by seismic work. Six producers have been completed with an aggregate potential of about 1000 b.d.

Twenty-nine tests were drilled; sixteen were dry.

Tables give data on the five fields and summarize the findings of the 1949 wildcats. G. D. H.

365. Oil and gas developments in south central Texas during 1949. W. H. Spice. *Petrol. Tech.*, July 1950, **2** (7), 76–82.—552 wells were drilled in 1949, 238 being wildcats. 242 oil wells were completed, eleven opening new fields. There were seven gas and gas-condensate wells. Big Foot is a new Navarro sand find. Marsha, Southwest Byersville, and South Coupland are Dale limestone finds. Lund is a serpentine intrusion.

The 1949 production of 9,500,084 brl came from 3568 producers. 46% of the oil was from the old Edward limestone fields—Luling, Branyon, Salt Flat, and Darst Creek.

Tables list the fields with data on oil and gas production, numbers of wells, producing formation, etc., and give information on outstanding 1949 wildcats. A map is attached. G. D. H.

366. Oil and gas developments in north central Texas during 1949. J. B. Harrell. *Petrol. Tech.*, July 1959, **2** (7), 65–75.—2288 wells were drilled in 1949, 970 giving oil and 524 gas. One of the most important discoveries was the Pardue field in Fisher County. The Humphrey Grey discovery is also important.

Tables give the stratigraphic sequence, a list of the important discoveries, data on important successful wildcats, and a list of the fields with data on production, producing horizons, deepest formation tested, etc. G. D. H.

367. New oil and gas fields of Western Canada. K. M. Fagin. *Petrol. Engr*, Oct. 1950, **22** (11), B-7.—The rate of development of the Leduc–Woodbend and Redwater fields is expected to decline in 1951 as productive limits are now known. Elsewhere in Western Canada development is expected to increase. The new pipeline from Edmonton to Lake Superior will open a large market.

Since the discovery of Leduc in 1947, exploration has been concentrated in Alberta, but future exploration is expected to cover the whole of the 800,000 sq. miles sedimentary basin in Western Canada.

Three new Devonian reef fields have been found this year. In the Acheson field, the discovery well, 8 miles west of Edmonton, found 121 ft of D_3 pay at 4969 ft. Production may be developed from the D_2 and Cretaceous sands. In the Camrose field, the discovery, Flint 1, on test produced about 700 b.d. from a 30-ft D_2 pay. Productive limits of the field are not known. Valley 7, which discovered the Big Valley field, 80 miles northeast of Calgary, on test flowed oil and gas from a D_2 pay at 5237–5252 ft.

The Redwater field, the largest in Canada, has 800 D_3 producers. Initial production averages 2000 b.d. with G.O.R. 215 : 1. Max pay thickness is 207 ft. The D_2 has not been found productive. The D_3 is being developed on the southeast and northeast parts of the field. Ultimate recovery is estimated at 320–640 million brl, depending on the efficacy of the water drive.

The Leduc–Woodbend field, 16 miles southwest of Edmonton, discovered in 1947, has 450 producers in the D_2 and D_3, and Lower Cretaceous sand. D_3 and D_2 are twinned on 40-acre spacing when both zones are productive. The productive area is about 25,000 acres and total field development will require about 900 wells. Estimated reserves are 375–500 million brl. Cumulative production to date is about 20 million brl.

Over forty companies are operating in Western Canada. Seismic, gravity, and magnetometer work is in progress.

Drilling and completion practice in the Redwater and Leduc fields are briefly described. C. A. F.

368. The Redwater oil field. J. V. Drumheller. *Petrol. Engr*, Oct. 1950, **22** (11), B-50.—The Redwater field, Alberta, was discovered by Redwater 1, completed in 1948. Approx 600 wells are producing, and completion rate is approx ten wells/week. On present 40-acre spacing, a total of 950–1000 wells will be required. Most wells are now on pump.

Pay is Upper Devonian reef limestone at about 3250 ft. There is no gas cap, and the reservoir fluid is undersaturated. Porosity averages 8% and pay averages 88 ft. Virgin b.h.p. was 1050 p.s.i.g. and G.O.R. 140 : 1. Cumulative production to July 1950 was about 8,765,000 brl, and recoverable reserves are estimated at 600,000,000 brl.

Completion and drilling practice are briefly described. C. A. F.

369. The Schoonebeck oilfield in the Netherlands. W. Th. B. Reimering. *Petroleum*, Aug. 1949, **12** (8), 191–3.—A comprehensive review is given of the development of the oilfield, stated to be the largest in Europe. Geology of the area, including a typical well log, is given and its effects on spacing and locating of wells considered. The

review includes an account of drilling and production of the crude which is transported by rail tank cars to the refinery at Pernis.

The crude has a specific gravity of 24·8° A.P.I., contains 5–6% of paraffins but little gas, and has a max pour point usually between 43° and 54° F. A desalting stage is necessary, as the salt content of the crude after dehydration is too high for refinery requirements. R. E. P.

Transport and Storage

370. Tanker developments. R. Hammond. *Petroleum*, Oct. 1950, **13** (10), 249–51.—Reviews the most significant developments in the tanker transport field, including new rules for tabulating scantlings by Lloyd's Register, new designs and design considerations, marine fuels, and finally, lists tankers built in British yards during the year. R. E. P.

371. Oilfield transport. Anon. *Petroleum*, Oct. 1950, **13** (10), 255–7.—Specialized type of oilfield transport media are discussed and the history of the introduction of vehicles to the oilfields is reviewed. R. E. P.

372. Oil transport by road. Anon. *Petroleum*, Oct. 1950, **13** (10), 252–4.—The history and design of oil transport by road is reviewed. Transport regulations and tonnage limitations are discussed, and changes in the laws relating to these factors suggested. R. E. P.

373. Oil by rail and canal. Anon. *Petroleum*, Oct. 1950, **13** (10), 258–61.—A historical review of the use of rail and canal for the transport of oil products in Britain. Changes in regulations, and in barge and rail-car design through the years, are recorded. R. E. P.

374. Crude oil and refined products pipeline mileage in the United States. January 1, 1950. A. T. Coumbe and I. F. Avery. *U.S. Bur. Mines Inf. Circ.* 7585, Oct. 1950.—Mileage, diameters, etc., of trunk and gathering crude and product pipelines in use on January 1, 1950, are recorded for forty states, and the data are compared with those of earlier years since 1936. The data are discussed with respect to average diameters, mileage increase since May 1941, the larger-diameter trend and daily capacity for crude oils and gasoline lines, and the Government lines sold for natural-gas transport. The information shows: an increase of 11,800 miles since May 1941 for refined products and 6193 miles for crude oil, the total length for all lines being 152,463 miles. W. H. C.

375. Pipeline corrosion by sour crudes. L. R. Sheppard. *World Oil*, May 1949, **129** (2), 193.—Failures associated with extensive corrosion by crudes containing hydrogen sulphide or sulphur in other forms are frequently located only when the condition of the pipeline is critical. Various methods exist for the detection of extensive corrosion, but, except for the periodic inspection of the equipment, other tests are seldom truly representative of the conditions resulting from the corrosion.

Brines greatly increase the corrosive hazards, in particular in the presence of oxygen. The weakening of the metal is sometimes brought about by hydrogen "permeation."

Treatments have yet to be developed for the reduction of hydrogen sulphide, oxygen, brine, and sediment. The trends of such developments are discussed. A. J. H.

376. "Super-inch" gas line is complete. D. H. Stormont. *Oil Gas J.*, 9.11.50, **49** (27), A54.—The construction of the gas line from West Texas to San Francisco is reviewed. This is the world's largest-dia high-pressure natural-gas transmission line, and by the first of 1951 the line will be transporting 150,000 M.c.f. daily, and a year later will carry 400,000 M.c.f. daily into the Bay area. Cost was 63,300,000 dollars. G. A. C.

377. Gas-transmission-line maintenance. L. W. Curfman. *Oil Gas J.*, 30.11.50, **49** (30), 84.—The correct maintenance of gas-gathering and transmission lines is

vitally important, and for this an efficient foreman qualified for the job is necessary. The qualifications and scope of duties of such a man are described. A table shows number of foremen, line walkers, and gang men desirable for lengths of line from 200 to 2000 miles; a max of 200 miles is the optimum for each foreman. G. A. C.

378. Equation for optimum pipe size with viscous flow. E. Long. *World Oil*, Apr. 1949, **128** (13), 197.—The author approaches the selection of the optimum pipe size from the consideration of the "critical viscosity X," described for the purpose of this treatment as "the viscosity at which the total costs of two successive pipe sizes are the same," the total cost including pumping and initial pipe costs. Thus the larger pipe is selected for the practical case of a fluid the visc of which is greater than the above critical.

The flow and cost equations are combined for the evaluation of the "critical viscosity," reducing to an equation involving the pipe size, and the cost and flow conditions. A specific example is taken.

References. A. J. H.

379. Radioactivity in pipeline flow studies. D. E. Hull, J. W. Kent, and R. D. Lee. *World Oil*, May 1949, **129** (1), 187.—The use of radioactive barium, in the form of an oil-soluble soap, enabled the distribution of the interface between two stocks to be traced.

The method of radioactive tracer study is relatively cheap and simple in operation, and may assist in the diversion of stocks arriving at a point through a single main line. A. J. H.

380. Economics of a products pipeline. R. H. Lynch. *Oil Gas J.*, 16.11.50, **49** (28), 214.—The article summarizes the data and studies that should be obtained preparatory to making a decision to build a products pipeline.

Lay-out, costs, and return on invested capital are discussed. The article is illustrated by thirteen figures. G. A. C.

381. Explosion-proof motors in pipeline operations. F. G. Geiger. *World Oil*, Apr. 1949, **128** (13), 195.—A brief report on the advantages of electric power for use in pipeline installations, and of the necessity of having means of reducing explosion hazards. Two preventive methods are available: the isolation of the electric motor from the centrifugal pumps, by means of partitioned chambers, and the use of explosion-proof motors. The construction of a typical motor is explained. A. J. H.

382. Pulsating pressure plant. Anon. *Mech. World*, 3.11.50, **128** (3329), 430.—The paper describes a new pulsating pressure plant recently installed in the pressure-vessel laboratory of the British Welding Research Association. It is to be used for testing vessels by repeated applications of pressures. This type of test has a number of advantages over the normal static pressure test, the chief of which is the fact that vessels which will be subjected to varying pressures in service can be tested under similar conditions. The plant is described, a number of illustrations are given, and the main features are described. The main pump is a three-ram pump capable of a delivery of 12 g.p.m. against a pressure of 6000 p.s.i. and is driven by a 75-h.p. electric motor. There is also a small pump capable of a delivery of 1 g.p.m. against a pressure of 6000 p.s.i. and an air compressor with an intake of 40 cu. ft/min. A. S.

383. Pressure surges in pipelines. E. T. Skinner. *World Oil*, May 1949, **129** (1), 196.—Pressure surges encountered are due to too rapid closing of valves, and to the interrupted fluid motion associated with reciprocating systems of pumping. They may cause increases of as much as double the normal operating pressures.

Prevention of the dangers includes the more careful operation of valves, and the installation of by-pass pipes or surge tanks. The main disadvantage of the latter is the rate at which the gaseous elastic medium is absorbed, if a closed tank, or the high evaporation losses encountered with open tanks.

The appendix gives a mathematical approach to some of the surge factors.

References. A. J. H.

384. Expansion of "L" type bends. F. E. Wolosewick. *Refiner*, 1950, **29** (9), 143–4.—The simple L-type bend, often called right-angle bend, is also useful for providing for thermal expansion. Simplified graphs are presented for calculating equilibriating forces and movements due to thermal expansion. For more precise values a set of equations is suggested. An illustrative example is given. R. W. G.

385. The measurement of the thermal conductivities of soils, and the calculation of oil temperatures in buried pipelines. K. G. Brummage. *J. Inst. Petrol.*, 1950, **36**, 575–92.—The temp of the oil in large-dia buried pipelines is important in the following connexions :—

(1) Through its effect on visc it controls the throughput.

(2) It is sometimes possible to cool the engines at pumping-stations by heat exchange with the oil being pumped, and the size of the heat exchanger depends on the oil temperature.

(3) The protective coating on the pipe must be chosen to withstand the pipe temp without deformation.

The oil temp is determined by the thermal conductivity and the temp of the soil. In this paper measurements upon existing buried pipelines are described to determine these quantities under summer conditions at various places in the Middle East. The theoretical analysis is given, including allowances for the insulating effect of pipe-coatings. A simple procedure of general application is then described for calculating the temp of liquid in turbulent flow in a buried pipeline. A. R. W. B.

Refinery Operations

Refineries and Auxiliary Refinery Plant

386. Altona refinery of the Vacuum Oil Company. Anon. *Chem. Engng and Min. Rev.* (*Australia*), 10.8.50, **42** (11), 127.—Brief details and five illustrations are given of the Altona refinery of the Vacuum Oil Co., at Victoria, Australia. The plant processes New Guinea crude and other crude oils and produces all grades of products, including lubricating oils and bitumens. The plant's centralized automatic control system and its operation, and the treatment of water before its discharge into the Kororoit Creek, are outlined. W. H. C.

387. British refineries (No. 8). The Shell Haven refinery of the Shell Refining and Marketing Co. Ltd. Anon. *Petroleum*, Sept. 1950, **13** (9), 224–7.—Describes the new refinery which is being built at the mouth of the Thames, and which is designed to process crude oil from the Middle East.

Cuts obtained by distillation of crude are light and heavy gasoline, light and heavy naphthas, gas oil and residue. The straight-run gasoline will be passed through a doctor-treating plant and a tetra ethyl-lead-blending unit to increase the octane number to 70 or 80. Naphtha will pass through a reforming unit, the doctor plant, and tetra-ethyl-lead-blending unit to obtain high-octane aviation fuel.

Cooling water will be obtained by tunnel from the Thames, and wide provision has been made for automatic control of the plant. R. E. P.

388. Lube unit being added to carbon plant by Shell. Anon. *Refiner*, 1950, **29** (9), 131–2.—General description of the Venezuelan Oil Concessions Ltd. (Shell's) Cardon refinery in Venezuela. This refinery was started in 1945, and first went on stream in February 1949. Since then the vacuum unit, the cracking units, the reforming unit, the gas-recovery units, the polymerization unit, and others have been put in operation. Now a comprehensive lubricating-oil complex, comprising a de-asphalting unit (2560 b.d.), a furfural extraction unit (63520 b.d.), solvent dewaxing unit (3520 b.d.), and clay-contacting unit (3700 b.d.) is being constructed. This should be completed by the second half of 1951 and will yield 2000–2500 b.d. of high-grade lubricating oils. R. W. G.

389. Test run gives data on T.L.L. commercial operation. Anon. *Refiner*, 1950, **29** (9), 115–20.—A detailed report of the two-week test run carried out on the one-

year-old T.C.C. at the Socony-Vacuum Oil Co.'s refinery at Casper, Wyoming. During the test run all distillation and cracking facilities were operated under uniform conditions, corresponding to normal refinery operations. As is normal refinery practice, sweet green crude was charged to one crude still and sour black crude to the other. Flow diagrams, compositions of products and complete material balances are given. It is reported that the check run proved to be quite satisfactory. R. W. G.

390. Furnace development in the refining industry. R. Dolez. *Bull. Ass. franç. Tech. Pétrole*, 1950 (84), 45–54.—Progress from the shell still to the modern furnace having both radiation and convection zones is outlined. Fuel consumption, for equivalent heat input, has been reduced to 30% of the former figure, whilst heat-transfer rates have greatly increased, attaining values of 100,000 k. cal./hr/m² in experimental designs, *i.e.*, over twelve times the value for a shell still. In modern furnaces the importance of the radiant zone has increased at the expense of that of the convection zone which latter, particularly in cracking furnaces, is being largely replaced by air pre-heaters. A vertical cylindrical furnace design is economical in construction costs, but heat-transfer rates are limited to approx 55,000 k. cal./hr/m². V. B.

391. Planning heat transfer equipment. Part I: Basic relations. T. G. Hicks. *Refiner*, 1950, **29** (9), 97–101.—The series is intended to be of assistance to refiners making their own preliminary heat-exchanger calculations before submitting actual requirements to plant designers. Part I consists of an elementary presentation of the general heat-transfer equations for exchangers involving the use of log mean temp differences, varying overall heat-transfer coeff, film, and scale coeff, and correction factors for multipass heaters. Consideration is given to all five types of heat exchange, *i.e.*, constant hot fluid temp, constant cold fluid temp, simultaneous constant hot and cold fluid temp, and parallel and counter-current flow exchangers.

Two illustrative examples are worked out. R. W. G.

Distillation

392. Packaged gasoline plant is completely portable. L. S. Gregory. *Oil Gas J.*, 30.11.50, **49** (30), 60.—The plant described is installed at Garrison Pool, Oklahoma, and permits a producer with a small gas volume to recover gasoline economically, and L.P.G. if required, without hazard of large investment. 600,000–700,000 cu. ft/day is processed. Tables show yield data, and plant production and sales data for a period in 1950.

Replacement value is approx $30,000, and labour costs average 1 hr/day. G. A. C.

393. Bubble trays—design and layout. Part II. J. A. Davies. *Refiner*, 1950, **29** (9), 121–30.—Paper presented to enable relatively inexperienced plant process personnel to design satisfactory trays. Part II considers actual tray lay-out calculations and design in detail, including general reference to effect of various factors on tray efficiencies. Equations and recommended values for the following specific items are given: liquid gradient, v.p. drop, downflow free height, downflow residence time, downflow width, max slot velocity, vapour-distribution ratio, vapour distribution per section, and tray spacing. Approx number of caps given by superficial slot velocity of $12{\cdot}1/\sqrt{\rho g}$, where ρg is the vapour density in lb/cu. ft. Tray balance, flexibility, and stability are also considered, and various possible tray flow patterns indicated.

An illustrative tray calculation is given for a typical lay-out. A corrected diagram for tray draw off as given in Part I, is presented. R. W. G.

Absorption and Adsorption

394. Plymouth's operations at Benedum. R. F. Carlson. *Oil Gas J.*, 23.11.50, **49** (29), 66.—Production practices of the Plymouth Oil Co., Texas, storage facilities, and separation system dealing with the 28 million cu. ft. of gas from twenty-nine completions in the Benedum field are described. Three-stage separation is used, distillate production is 4000 brl; storage is accomplished in one large battery of thirty tanks. G. A. C.

395. California nation's second-ranking state in natural-gasoline-plant capacity. Anon. *Oil Gas J.*, 9.11.50, **49** (27), A.59.—Natural gasoline and cycling-plant operations in California are listed for June 1950. Its seventy-four gas-processing plants have a daily output capacity of 4,685,000 gal of liquids. This capacity is second only to Texas. G. A. C.

396. Gasoline plant features combination cooling tower and air-cooled condenser installation. Anon. *Oil Gas J.*, 23.11.50, **49** (29), 71.—Units of the gas-processing plant of the Plymouth Oil Co., Texas, which deals with the gas from the Benedum field, are described.

Owing to scarcity of water, it was necessary to use air-cooled condensers and coolers where practicable; the air is drawn through a cooling tower where the limited amount of water used in the plant is cooled simultaneously with the air flowing through the tower. G. A. C.

397. Product requirements determine the complexity of gas recovery processes. C. C. Pryor. *Refiner*, 1950, **29** (9), 204–14.—The development of markets for natural gasoline and light hydrocarbons continues to control natural-gasoline process-plant technology. Besides natural gasoline for refinery blending and propane and butane for "bottled gas," natural-gasoline plants produce light hydrocarbon products for petrochemical industry. Some plants produce finished motor gasoline, which may have been reformed to bring up to premium standard.

Gas-recovery processes and process methods are discussed. While the fundamental process steps used for recovery of liquefiable hydrocarbons—absorption, stripping, and fractionation—are the same in all plants, methods vary due to such conditions as scarcity of cooling water, uncertain supply of electricity, wet or dry raw gas at high or low pressure, and whether residue gas is to be sold or recycled to a formation. Recent development is the use of fractionating-absorbers for the removal of methane and ethane at primary point of contact with lean oil. R. W. G.

398. Hypersorption process produces up to four separate product streams. Anon. *Refiner*, 1950, **29** (9), 216.—The hyposorption process, developed by Union Oil Co. of California, Los Angeles, is briefly described. Hypersorption is the separation of light gases by continuous adsorption on charcoal. The adsorbant falls by gravity through the hypersorber, retaining the heavier gaseous components, which are later stripped off by steam. The regenerated charcoal is transferred to the top of the hypersorber by gas lift. Hypersorption is suitable for recovery of ethylene and propylene from refinery gases and high-temp cracking gases, separation and purification of hydrogen, recovery of acetylene in low concentration gas streams, recovery of ethane and propane from natural gas, and the separation of nitrogen from natural gas. R. W. G.

399. Temperature entropy chart of thermodynamic properties of nitrogen. E. S Burnett. *U.S. Bur. Mines Rep. Invest* 4729, Sept. 1950.—The temp entropy (T,S) chart of thermodynamic properties of nitrogen presented, prepared for use in the helium section of the Bureau of Mines, is a considerably extended revision of the information published in 1949. The data and method of its preparation are described, and twenty-four references, together with twelve references to the Leiden laboratory communications on P-V-T. and other data, are included. W. H. C.

Solvent Extraction and Dewaxing

400. Furfural's role in middle distillate problem. M. Halpern. *Oil Forum*, Nov. 1950, **4** (11), 435.—The amount of low-sulphur, high-quality straight-run distillate available is far less than the amount required to meet even current demands, and the demands for the products falling within the middle distillate category are increasing rapidly. These demands come from jet aircraft, diesel engines, and domestic heating appliances, and supplies are also required for catalytic-cracking operations. Means must be provided whereby nitrogen and sulphur contents and certain aromatics and other constituents are reduced to a tolerable level. A number of processes can provide these improvements, but most of them have the disadvantage of high cost. However, furfural extraction provides a single processing step for upgrading an extremely wide

range of middle distillate stocks. In addition, it is sufficiently flexible to produce high-quality gasolines, furnace oils, and diesel fuels in varying amounts as required by the market situation. A description is given of the new Eagle Point refinery near Camden, N.J., in which the furfural process plays an important part. A. S.

Cracking

401. Autothermic cracking for ethylene production. R. M. Deanesly. *Refiner*, 1950, **29** (9), 217.—In this process part of the charge gas (ethane, propane, etc.) is burnt with air or O_2 to provide the high temp desirable (1700°–1800° F). The endothermic reaction reduces the reactor effluent gas to 1500° F. The gas is cooled to remove water, aldehydes, and traces of acid, passed over a hydrogenation catalyst to hydrogenate acetylene and any butadiene. The gas is then cooled and washed with alkaline water. Optimum conditions are 80% conversion; this gives max concentration of ethylene in product. The use of heat exchangers to preheat charge reduces quantity of air or O_2 to be injected to reactor to such an extent that N_2 dilution due to air is of little importance, making O_2 economically undesirable. R. W. G.

402. Economics of petroleum coking for catalytic cracking feed preparation. J. W. Jewell and J. P. Connor. *Oil Gas J.*, 23.11.50, **49** (29), 64.—Delayed coking process is evaluated relative to each of the parallel and competing vacuum flashing, vis-breaking, propane decarbonizing, and combination methods.

Summaries and figures indicate that the delayed-coking process enjoys superior earning power in a soft fuel-oil market or a good coke market. Feed stock and type of operation constitute principal quality-determining factors rather than process itself; and an unlimited market and periodic fuel-oil problems indicate a place for the process for many years. G. A. C.

403. Furfural process adapted to operate on wide variety of charge stock. Anon. *Refiner*, 1950, **29** (9), 196–8.—The furfural solvent-extraction technique for lubricating oils has been extended to refining diesel oils. Reference is made to the Westville, N.J., furfural plant, which was designed to charge 8000 b.d. of low-boiling distillates. This plant has been in commercial operation since December 1949 processing a wide variety of stocks. The process, due to its aromatic, sulphur, and nitrogen selectivity, is particularly suitable for diesel and furnace oils. Tabulated data are presented showing properties of processed gas oils, which have improved ignition properties. Furfural refining of cat-cracking charge reduces lay-down on cracking cat, improves cat activity, reduces hydrogen factor obtained, and leads to improved O.N. of leaded finished gasolines. Furfural also selective towards removal of metal contaminants—iron, vanadium, and nickel. R. W. G.

404. New Houdriflow installations employ modified design. Anon. *Refiner*, 1950, **29** (9), 170–5.—A discussion of the recent modifications to the Houdriflow design, which include: (1) elimination of intermediate seal leg between reactor and kiln which simplifies vessel design and effects reduction in unit height, (2) elimination of pressure balance between reactor and kiln such that pressure at top of kiln is essentially atmospheric, (3) new simplified type of catalyst and vapour disengager, (4) use of relatively inexpensive thermo-compressions to compress the portion of the kiln flue gas utilized in the catalyst lift. The Houdriflow design, which was first announced two years ago, is similar to the T.C.C. process, except that the cat regenerator kiln is superimposed on the reactor and cat gas lift is employed in favour of bucket elevators. A list of commercial Houdriflow installations is given. R. W. G.

405. Platforming—new continuous catalytic reforming process operates on all types virgin stocks. J. F. Wright. *Refiner*, 1950, **29** (9), 163–7.—Platforming is a recent continuous (high-liquid volumetric recovery) cat reforming process using platinum catalyst in a hydrogen atmosphere, suitable for straight-run, natural, and cracked gasolines. O.N. improved by: (1) dehydration of naphthenes to aromatics; (2) hydrocracking of high-mol.-wt. paraffins; (3) isomerization of paraffins and of naphthenes; (4) cyclization of paraffins; (5) desulphurization. Properties of platformate include: (1) laboratory O.N. upgraded by as much as 50; (2) good lead susceptibility; (3) good road performance; (4) hydrocarbon composition, *iso*paraffinic and aromatic being

particularly suitable for coming high-compression automobile engines; (5) 90% or more sulphur reduction; (6) good stability, inhibitors only necessary to stabilize tetra-ethyl lead. R. W. G.

406. Some aspects of heterogeneous catalysis in the petroleum industry. E. W. Nagelstein and A. Guillemin. *Rev. Inst. franç. Pétrole*, 1950, **5**, 355–62.—Table is given showing main cat reactions used in the petroleum industry; this is followed by a summary of the recent symposium on aromatization cat organized by the I.F.P. Report is in four sections, dealing with aluminas intended as cat supports: (preparation, activation (optimum temp 700°–750° C), physical examination), the cat themselves, (preparation, composition, physical examination), the cyclization reaction, and suggestions for further work. V. B.

407. Fluid catalytic cracking process improved through better design of components. R. T. Armstrong. *Refiner*, 1950, **29** (9), 190–4.—A discussion of some of the features of fluid catalytic-cracking equipment, including: (1) catalyst recovery, (2) catalyst stripping, (3) catalyst carry-over from reactor, (4) catalyst handling, and (5) regenerator lining. A brief discussion is also given of the general design of the modern downflow fluid cat cracker. Commercial fluid cat units to-day remain on stream at high throughput rates for long periods, often over a year, as a result of the attention devoted to individual details of equipment design and choice. R. W. G.

408. T.C.C. units designed for small refineries. Anon. *Refiner*, 1950, **29** (9), 183–4.—Southwestern Engineering Co., Los Angeles, and the Socony-Vacuum Oil Co., New York, have jointly designed small cracking units, based on the Thermofor catalytic cracking. The units cover a range of 1000–8000 b.d., and require a limited entire site of about 150 by 200 ft, thus being particularly suitable for use in improving gasoline product by small refineries. The units consist of three essentials: (1) feed preparation, (2) catalytic cracking, and (3) stabilization. A unique feature of the catalytic section is the utilization of a standard-type field derrick to support the various T.C.C. components. The whole of the unit is shop fabricated for final assembly on the site. R. W. G.

409. Gravity separation of inactive catalyst. C. K. Viland. *Oil Gas J.*, 30.11.50, **49** (30), 74.—After experiments in a laboratory, a gravity-classification system based on the change in density of a catalyst with age has been installed at the Avon refinery of the Tide Water Associated Oil Co., California.

Essentially the equipment consists of a centrifugal separator to separate a fine and coarse fraction of the catalyst, followed by a classification table which rejects denser particles from the coarse fraction. The fines from the centrifugal separator and the lighter particles from the gravity classification table are returned to the catalytic-cracking unit for further use.

The unit is believed to be the first commercial application of the discarding of higher-density and lower-activity fluid-type catalyst from a mixture. Tables shown include a test on catalyst-classification equipment, physical characteristics of two component mixtures, and comparative properties of fresh fluid-type catalyst. G. A. C.

410. Socony-Vacuum T.C.C. units move catalyst by gas lift. A. V. Danner. *Refiner*, 1950, **29** (9), 179–82.—A brief description of the 1951 design of the Socony-Vacuum T.C.C. unit. One of the principal innovations is the use of cat gas lift instead of bucket elevators. Reference is made to the "package" T.C.C. design, which is for "total charge capacities" below 6000 b.d. Design is similar in detail to the general design for larger units, but the whole of the unit may be prefabricated and merely erected on site. R. W. G.

Chemical and Physical Refining

411. Gas-treating process. A. L. Kohl. *Oil Gas J.*, 30.11.50, **49** (30), 63.—Problems, principal contaminants, and processes utilized in gas treating are reviewed.

Principal contaminants include H_2S, CO_2, and H_2O vapour, lesser ones are mercaptans, CS_2, carbon oxysulphide, O_2, and N.

H_2S can be removed by processes in which absorbent is regenerated by oxidation of sulphur compound to free sulphur, or in which H_2S is stripped from the absorbent unchanged by application of heat or stripping vapour.

Figures show flow arrangement for the iron oxide dry box and the Thylox gas-purification systems.

The Seaboard sodium carbonate process is an example of the heat-reactivated type; and a flow diagram is given with reaction equations, for the tripotassium phosphate and glycol-anine gas-treating processes.

Processes based upon the use of ethanolamine solutions, including mono-, di-, and tri-ethanolamines, are widely used for treatment of natural and refinery gases.

G. A. C.

Special Processes

412. Acetylene manufactured by electric discharge in Schoch process. L. S. Daniels. *Refiner*, 1950, **29** (9), 221–4.—A description of the method which is now at the planning stage of commercial development. In this process methane or higher hydrocarbon gases or liquid petroleum (gasoline to diesel fuel) are used as charge. Reaction takes place in the discharge chamber where electric discharges provide heat for pyrolysis. To prevent damage to electrodes, the average discharge chamber temp does not rise above 550° F. Small amounts of carbon black produced are removed by cyclones and bag filters from reactor products. C_3 and heavier hydrocarbons are separated from acetylene by normal absorption processes. The acetylene is then absorbed by dimethoxy-tetra-ethylene-glycol ("D.M.T.G.") to separate from hydrogen, methane, ethane, and ethylene. The C_3 and heavier hydrocarbons are recycled. It takes about 1·6 lb of gas or 2·15 lb of liquid hydrocarbon to produce 1 lb of acetylene. R. W. G.

413. The fluidized-solids technique and its industrial applications. C. C. Hall and L. J. Jolley. *Petroleum*, Sept. 1950, **13** (9), 217–23.—Summarizes the characteristics of fluidized solid systems, indicating their advantages and limitations, and reviews their applications to industrial processes. These include catalytic cracking, Fischer–Tropsch synthesis, manufacture of phthalic anhydride, carbonization and gasification of solid fuels, retorting of oil shale, lime burning, hydrogenation of coal, and town-gas purification.

Technically important features of fluid-bed systems are: (*a*) uniformity of temp, (*b*) high rates of heat transfer, (*c*) uniformity of composition of the solid phase, (*d*) reduced effective thickness of the stagnant gas-film at the surface of the particles, (*e*) the possibility of circulating solid continuously between two or more separate reactors in which the solid is in contact with different gas phases, (*f*) the possibility of using the solid phase as a thermophore conveying heat to or from the reactor, and (*g*) the possibility of continuously renewing a portion of the solid phase or of altering its composition at will without affecting its homogeneity. Thus fluidized-bed methods are suitable for processes in which close control of temp must be maintained in spite of large heats of reaction. High rates of reaction possible are due to the large surface rather than due to inherent superiority of fluidized conditions. R. E. P.

414. Low molecular weight olefins from petroleum as raw material. M. Ruhemann and H. Steiner. *Petroleum*, May 1949, **12** (5), 112–16.—Reviews the special processes which have been developed for the production and purification of low-mol.-wt. olefins from petroleum. The primary object is to produce a max amount of ethylene with a minimum of hydrogen, paraffinic gases, and liquid products. Conditions necessary are generally high temp, low pressures, and short contact times. Various processes are briefly described, including the Catarole and Thermofor pyrolytic cracking processes, which also produce aromatics by aromatization of liquid residues.

Separation and purification of the individual olefins from the cracking gases is also considered. Fractional distillation may be used to separate olefins into groups, but the production of pure components from the groups is more difficult. Four methods of separation are described: (*a*) reversible chemical combination; (*b*) absorption in liquid solvents; (*c*) fractional sorption and desorption from solid adsorbents; and (*d*) partial condensation followed by fractional distillation. The last method is used in the Catarole plant, and is treated in greater detail. R. E. P.

415. Progress in petrochemistry—a technical report. W. J. Sweeney. *Oil Gas J.*, 2.11.50, **49** (26), 74.—Modes of preparation of chemicals from ethylene, propylene, and butylenes are given, and uses of various products indicated.

Figures show hydration, chlorination, oxidation, hydrochlorination, alkylation, and polymerization steps in the syntheses. The commercial production of synthetic rubbers and their properties are dealt with, and possibilities of using refinery streams for preparation of chemical compounds for sale as such to chemical manufacturing concerns reviewed.

An example of the latter is petroleum *ortho*-xylene, which may be used with coal-tar naphthalene for preparation of phthalic anhydride; the xylene being produced along with other aromatics in the "hydroforming" process for converting heavy naphtha factors into high-octane gasolines.

Other petrochemical products include naphthenic and cresylic acids. G. A. C.

416. Chemicals from natural gas and petroleum. G. Egloff. *Oil Gas J.*, 2.11.50, **49** (26), 69.—A general review of the manufacture of chemicals from petroleum products is given. Less than 0·5% of total crude oil and natural gas production is used as base to produce over 3000 million lb chemical compounds.

Cracking of oil and gases; dehydrogenation, polymerization, alkylation, and separation processes; preparation of derivatives by halogenation, sulphonation, nitration; manufacture of organic acids, alcohols, glycols, glycerin ethers, esters, aldehydes, ketones, halogen and sulphur derivatives, and nitrogen compounds are dealt with.

Expansion of the petrochemical industry in the U.S. and Great Britain is briefly reviewed.

It is stated that the known half a million organic compounds can be synthesized from petroleum. G. A. C.

417. Magic of petrochemistry opens new horizons for oil and gas. Anon. *Oil Gas J.*, 2.11.50, **49** (26), 51.—A brief review of the plants and operations of Phillips Chemical Co. at Etter, Borger, and Houston is given. Anhydrous ammonia, nitric acid, ammonium nitrate, ammonium sulphate, butadiene, copolymer, and furnace blacks are items dealt with. G. A. C.

418. Production of hydrocarbon feed stocks for petrochemical processes. M. van Winkle. *Refiner*, 1950, **29** (9), 226–38.—General review of the methods of preparation. With the exception of benzene, all of the primary hydrocarbon and associated components necessary for petrochemicals are obtainable from natural gas, natural gasoline, and refinery cracked gases. Benzene may be obtained from petroleum aromatization processes. Pretreatment of gas includes desulphurization and possibly dehydration. Primary separation is afforded by absorption and secondary separation by distillation–low temp fractionation (at − 100° F) used where high ethane recovery offsets cost of refrigeration. The conversion processes of interest to the petrochemical industry are those which produce acetylene, olefins, and diolefins: (1) cracking-thermal, regenerative and catalytic, (2) catalytic dehydrogenation, and (3) Schoch electric-discharge process for acetylene production. R. W. G.

419. Sulphur produced from hydrogen sulphide. Anon. *Refiner*, 1950, **29** (9), 225.—An improved method. Many natural gas, refinery gas, and liquid hydrocarbon purifying operations yield by-product H_2S. The original Claus process for sulphur recovery utilized the reaction :—

$$H_2S + \tfrac{1}{2}O_2 \rightarrow S + H_2O.$$

This reaction is highly exothermic, and to give better control, the described process splits up the reaction into :—

(1) $H_2S + 3/2O_2 \rightarrow SO_2 + H_2O$;
(2) $2H_2S + SO_2 \rightarrow 3S + 2H_2O$.

Reaction (1) is carried out in a steam generator, and reaction (2) in a one- or two-stage converter. Single-stage converters give 80–85% sulphur recovery, and two-stage converters 90–95% recovery. R. W. G.

Metering and Control

420. Petroleum metering instruments. Anon. *Petroleum*, July, 1949, **12** (7), 175–6.—A very brief account of the importance and types of metering instruments. Describes the operating principles of the Tylor rotary-piston positive-displacement meter; the Rotameter flow meter, for continuous and direct reading of flow of a gas or liq in a pipeline; and measurement of liq flow by orifice plate and Venturi tube. R. E. P.

421. Pneumatic refinery instruments. Anon. *Petroleum*, Apr. 1949, **12** (4), 92–5.—Reviews some applications of pneumatic instruments in the petroleum-refining industry. Instruments are used to control pressure, temp, rate of flow, and liq level. Describes general principles, and gives diagrams and explanations of some instruments, including :—

(1) A liq-level transmitter designed to give accurate measurement of liq level. Accuracy ± 1 inch over range 12–450 inches applied liquid head.

(2) A two-position differential gap-controller used to meet process requirements where proportional action is undesirable, or to reduce wear on a valve or pump resulting from continuous or frequent operation.

(3) The Kent Mark 20 pneumatic controller, which operates with a wide range of diaphragm-operated control valves or with power cylinders for operating flue dampers, vanes, and induction regulators.

Also describes the *metavane* pneumatic distance transmission system and the application of the reset free-vane flow controller for the control of steam-turbines in the oil industry. The apparatus varies the speed of the turbine which operates a centrifugal hot-oil pump to maintain a predetermined rate of flow of oil from the pump.
R. E. P.

422. Graphic panels in operation. W. R. Jensen. *Refiner*, 1950, **29** (9), 139–42.—A discussion of the recently popular graphic instrument panels for process-plant control. These panels have been made possible by new small instruments suitable for mounting within a flow diagram. Principle advantages of this type of panel are : (1) operator can see all important process variables and their inter-relationship, with the result of ease of control; (2) refinery managers, etc. (who see the control board infrequently) can readily comprehend operation; (3) new operators understand entire process in less time; (4) control room is smaller and cheaper than conventional type; (5) remote-set controllers provide improved flow control. A list of nine plants employing graphic control panels in July 1950 is given, and two of these are mentioned more specifically. R. W. G.

Products

Chemistry and Physics

423. Determination of mechanism of catalyzed gaseous reactions. K. H. Yang and O. A. Hougen. *Chem. Engng. Progress*, Mar. 1950, **46** (3), 146–57.—The general procedures in planning and correlating experimental data on gaseous reactions catalysed by solids are surveyed. Methods are presented for eliminating, minimizing, or evaluating the temp and concentration gradients in gas films and catalyst pellets, and a summarized procedure is given for setting up the rate equations for many postulated mechanisms. J. G. H.

424. Catalytic hydrocondensation of carbon monoxide with olefins. 3. Polymerization and hydropolymerization of ethylene under conditions of hydrocondensing catalysis. Ya. T. Eidus, N. D. Zelinskii, and K. V. Puzitzkii. *Izvest. Akad. Nauk S.S.S.R., Otdel. Khim. Nauk*, 1950, 98–107.—Studies were made on the polymerization of C_2H_4 and the hydropolymerization of C_2H_4 and H_2 using the same conditions (190° C, atm pressure) and cat as for hydrocondensation of CO and C_2H_4. Using fresh cat no polymerization of C_2H_4 alone was obtained, but with H_2 a certain degree of hydropolymerization occurred as well as reduction of C_2H_4 to C_2H_6. When, however, a cat

already used for hydrocondensation of CO and C_2H_4 was employed products were obtained indicating occurrence of hydrocondensation of C_2H_4 with CH_2 radicals present on the cat. Reaction mechanism is discussed, and it is shown that hydrocondensation of CO and C_2H_4 is a specific separate reaction and not the sum of two independent reactions, viz., the polymerization (or hydropolymerization) of C_2H_4 and reduction of CO by H_2. **4. Hydrocondensation of ethylene with carbon monoxide at low concentrations of the latter.** Ya. T. Eidus, N. D. Zelinskii, N. I. Ershov, and M. I. Batuev. *ibid.*, 377–85.—Previous studies on hydrocondensation of C_2H_4 and CO were carried out on mixtures $1CO : 2H_2 : 3C_2H_4$; present work was done with 4–7% CO in the feed gas under otherwise similar conditions. 80–98·5% of the C_2H_4 reacts, of which up to 75% undergoes hydrocondensation and the remainder reduction to C_2H_6. In the temp range 100°–250° C optimum results were obtained at 190°. Using equimolecular quantities of H_2/C_2H_4 with 4–7% CO, 27% of the liquid products are C_3–C_4, remainder have b.p. 27°–300° C, and consist of aliphatics containing 70% unsaturateds. Examination (Raman spectra) of this material after hydrogenation indicated presence of *iso*-paraffins in the C_5–C_6 range. V. B.

425. On the influence on flame propagation of conditions at the longitudinal limits. H. Guénoche. *Rev. Inst. franç. Pétrole*, 1949, **4**, 515–20.—Reduction in the size of the open end of a cylindrical tube containing burning gaseous mixtures causes decrease in flame vibration, and may cause flame speed to become uniform. Theory based on the consideration of acoustical impedance explains the phenomenon and values calc for the diaphragm dia, using Bernouilli's law as modified by the Lutz and Jouguet theory, are in good agreement with experimental values found for $C_2H_2 + 0{\cdot}25\ O_2$ mixtures. E. B.

426. Concerning certain assumptions in the calculation of burning velocities. H. Gaudry. *Rev. Inst. franç. Pétrole*, 1949, **4**, 521–9.—Pressure/time curves obtained for explosions in spherical vessels are used in the study of burning velocities. Method remains valid where combustion products undergo reversible dissociation. Equations are developed and examples given. E. B.

427. Influence of turbulence on the mechanism of combustion reactions in the gas phase. M. P. Delbourg. *Rev. Inst. franç. Pétrole*, 1949, **4**, 530–8.—Variation of rate of combustion reactions in gas phase was studied as a function of the turbulent gas flow. Rate of turbulent reactions depends solely, like the rate of burning, on the Reynolds No. characterizing the flow. E. B.

428. Free valency number and chemical reactivity in oxidation reactions. R. Daudel. *Rev. Inst. franç. Pétrole*, 1949, **4**, 509–14.—Reactivity of a position in a non-polar mol is indicated by the free valency No. and the reaction rate is $\propto$ thereto. Examples are given correlating free valency No. of C_2H_4, styrene, stilbene, and $\alpha\alpha$-diphenyl ethylene with rate of oxidn by peracetic acid at 25° C. E. B.

429. Infrared spectrum of chlorofluoromethane. E. K. Plyler and M. A. Lamb. *Bur. Stand. J. Res. Wash.*, 1950, **45** (3), 204.—The infra-red spectrum of chlorofluoromethane has been measured in the gaseous state from 2 to 30 μ. The longest wavelength band was at 26 μ. Twenty-five bands were observed with cells of 5, 60, and 100 cm in length and gas pressures up to 60 cm of mercury. Nine of these bands are classified as fundamentals, and the remaining bands as harmonics and combinations. In the region of 1·5–2·5 μ several bands were resolved by means of a grating instrument into a series of lines. These bands are located at 1·657, 1·682, 1·700, 1·755, 2·229, 2·287, and 2·436 μ. The separation of the rotational lines for these bands is of the order of 2·3 cm^{-1}. J. T.

430. Composition and structure of di- and tri-*iso*butylenes obtained by the action of sulphuric acid on *iso*butyl alcohol. A. P. Meshcheryakov, M. I. Batuev, and A. D. Petrov. *Izvest. Akad. Nauk S.S.S.R., Otdel. Khim. Nauk*, 1950, 282–6.—A 1 : 1 mixture of *iso*butyl alcohol and 63% H_2SO_4 was refluxed for 6–8 hr. Product contained 30% C_8H_{16}, 40–45% $C_{12}H_{24}$, and 25–30% butylenes. Analysis of the liquid fraction by dist, hydrogenation, and Raman spectra showed di-*iso*butylenes to be

2 : 2 : 3- and 2 : 3 : 4-trimethylpentene. The tri-*iso*butylene (b.p. 176°–179° C) yielded, on oxidn, a monocarboxylic acid of m.p. 65°–67° C, thus identifying it as the tri-*iso*-butylene obtained by Butlerov (*J. Russ. Phys. Chem. Soc.*, 1882, **14**, 199) from *iso*-butylene, although the presence of other isomers is not excluded. In the hydrogenated product of the tri-*iso*butylene 2 : 2 : 4 : 6 : 6-pentamethyl-heptane was identified.
V. B.

431. Effect of ammonia in the feed gas on the catalytic hydrogenation of carbon monoxide to higher hydrocarbons. Ya. T. Eidus and I. V. Guseva. *Izvest. Akad. Nauk S.S.S.R., Otdel. Khim. Nauk*, 1950, 287–90.—A 1 : 2 CO : H_2 mixture was passed over CO cat at atm pressure and 190° C. NH_3 was added by bubbling the gas through conc NH_3 solution and drying over KOH. Addition of NH_3 caused inhibition of hydrocarbon formation, but poisoning effect of NH_3 on cat was easily reversible on withdrawing NH_3 from feed gas. No methylation of NH_3 was observed, but appreciable quantities of $(NH_4)_2CO_3$ were formed. Reactions occurring in presence of NH_3 are suggested.
V. B.

432. Influence of various types of hydrocarbon on the isomerization of 5-membered cyclanes in the presence of aluminium chloride. N. I. Shuikin, S. S. Novikov, and E. D. Tulupova. *Izvest. Akad. Nauk S.S.S.R., Otdel. Khim. Nauk*, 1950, 278–81.—Four series of tests were made with a 93°–156° C fraction from Baku gasoline using 5% of $AlCl_3$ at 80° C for 4 hr : (1) on gasoline which had been cat aromatized and then freed from aromatics, thus containing only alkanes (55%) and 5-membered cyclanes ; (2) on the unprocessed fraction, containing 9% aromatics and 40% six-membered cyclanes ; (3) on gasoline freed from naturally-occurring aromatics and containing 25·5% five-membered cyclanes, 43·5% six-membered cyclanes, and 31% alkanes ; (4) on fully aromatized gasoline containing 48·5% aromatics. Results indicate that isomerization is not affected by proportion of six-membered cyclanes present, but that a high aromatic content appreciably inhibits the reaction, probably due to formation of a complex between $AlCl_3$ and aromatic hydrocarbons.
V. B.

433. Isomerization of olefins and *cyclo*olefins in the presence of gumbrin. Kh. I. Areshidze. *Izvest. Akad. Nauk S.S.S.R., Otdel. Khim. Nauk*, 1950, 178–84.—Gumbrin, a naturally-occurring decolorizing clay, was used (1-cm spheres) for the simultaneous dehydration of alcohol and isomerization of the resultant olefin. *n*-butyl alcohol at space velocity of 0·08 litre/hr/litre cat at 450° C gave butene-1, butene-2, and *iso*-butylene ; *cyclo*hexanol at similar space velocity and 350°–400° C yielded methyl-*cyclo*pentene and *cyclo*hexene, together with small amounts of benzene and methyl-*cyclo*pentane. Dehydration, under similar conditions, of ethyl*cyclo*pentanol yielded methyl*cyclo*hexene, thus showing that gumbrin can cause both increase and decrease in No. of C atoms in the ring. Reaction mechanisms are suggested, and recent work (mainly Russian) in this field is briefly reviewed (25 references).
V. B.

434. Purification, purity, and freezing points of twenty-nine hydrocarbons of the API—Standard and API—NBS Series. A. J. Streiff, L. F. Soule, C. M. Kennedy, M. E. Janes, V. A. Sedlak, C. B. Willingham, and F. D. Rossini. *Bur. Stand. J. Res. Wash.*, 1950, **45** (2), 173.—The purification and determination of f.p.s and purity of the following hydrocarbons of the API–Standard and API–NBS series are described : 2 : 2 : 4 : 6 : 6-pentamethylheptane ; 1 : 1 : 2-trimethyl*cyclo*propane ; *cis*-2-hexene ; *cis*-3-hexene ; 2-methyl-1-pentene ; 4-methyl-1-pentene, 3-methyl-*trans*-2-pentene ; 4-methyl-*cis*-2-pentene ; 4-methyl-*trans*-2-pentene ; 4 : 4-dimethyl-1-pentene ; 4 : 4-dimethyl-*trans*-2-pentene : 2 : 3 : 3-trimethyl-1-butene ; *trans*-4-octene ; 1-nonene ; 1-decene ; 1-undecene ; 1 : 3-butadiene ; 1 : 2-pentadiene ; 1-*cis*-3-pentadiene ; 1-*trans*-3-pentadiene ; 1 : 4-pentadiene ; 2 : 3-pentadiene ; 2-methyl-1 : 3-butadiene (isoprene) ; 1 : 5-hexadiene ; 2 : 3-dimethyl-1 : 3-butadiene ; 4-ethenyl-1-*cyclo*hexene-(4-vinyl-1-*cyclo*-hexene) ; *cis*-decahydronaphthalene ; *trans*-decahydronaphthalene ; 2 : 3-dihydroindene (indan).
J. T.

435. Total ionization of hydrocarbons from mass spectral data. F. L. Mohler, L. Williamson, and H. M. Dean. *Bur. Stand. J. Res. Wash.*, 1950, **45** (3), 235.—The total ionization is calculated by adding all the mass peaks in the spectrum and multiplying by the sensitivity (current per unit pressure) for the max peak. This divided

by the corresponding product of *n*-butane gives a relative value independent of the units used. Data are taken from the A.P.I. Catalogue of Mass-Spectral Data. Total ionizations of isomers of a compound are nearly equal with only a few values differing significantly from the mean value. With some exceptions total ionization increases with increasing number of carbon atoms in each series $C_nH_{2n} + 2$, C_nH_{2n}, etc. Total ionization tends to decrease in some compounds with the same number of carbon atoms and decreasing number of hydrogen atoms, but there are numerous exceptions. Tables summarize data for 198 hydrocarbons. In some cases values of total ionization will permit computing all isomers as a group in chemical analysis without identifying the compound. J. T.

436. Thermochemistry for the petroleum industry. Part XIII.—Some oxygenated hydrocarbons, C_1 and C_2. K. A. Kobe and R. E. Pennington. *Refiner*, 1950, **29** (9), 135–8.—Data are tabulated for heat capacities, enthalpies, mean heat capacities, and heat-capacity equations (for a temp range of 0°–1200° C) for formaldehyde, acetaldehyde, methanol, ethanol, ethylene oxide, and ketone. The literature sources of data are given. R. W. G.

437. Viscosity data of organic liquids. J. W. M. Boelhouwer, G. W. Nederbragt, and G. Verberg. *Appl. Scientific Research*, 1950, **A2** (4), 249.—The visc of thirty-seven organic liquids with mol. wt. ranging from 120 to 807 have been measured. These consist of nine oxygen-containing compounds without ring structure, nineteen hydrocarbons with ring structure, six oxygen-containing compounds with ring structure, and three compounds containing S or I. The influence of ring and polar groups on visc is discussed. J. T.

438. Stabilization of carbon-black dispersions in hydrocarbons. M. van der Waarden. *J. Coll. Sci.*, 1950, **5**, 317–25.—This paper shows the way in which carbon-black dispersions in a hydrocarbon medium are stabilized by hydrocarbons.

Carbon black rubbed in vaseline forms a fine stable dispersion because the particles cannot move freely in the medium at room temp, but when heated the particles conglomerate into flakes. 0·1% dispersions were obtained by adding excess hydrocarbon oil to the cold vaseline–carbon dispersion. It was found that :—

(1) With aromatic-free hydrocarbons (white oil, etc.) pronounced flocculation was obtained, and the degree of flocculation decreased somewhat as the mol. wt. of the hydrocarbon increased. The same effect was observed when low-mol.-wt. aromatics were added.

(2) With alkylated aromatics and aromatic extracts in white oil stabilization was obtained, and was increased by heating to 100° C.

(3) With mineral-oil fractions (distillates, raffinates, extracts) stabilization was obtained only in the presence of high-mol.-wt. aromatics (*i.e.*, those containing side chains).

(4) The susceptibility of carbon black towards stabilization with aromatics is increased by removal of adsorbed volatile products from the carbon, but if the chemically-bound O_2 is also removed this increase is neutralized.

It is concluded that stabilization occurs by the adsorption of aromatics possessing a sufficiently long chain to prevent the particles from entering each other's sphere of attraction. H. C. E.

Analysis and Testing

439. Analysis of petrol–kerosine mixtures with specific reference to the boiling point. C. H. Manley. *Analyst*, 1950, **75**, 604.—The methods based on density and distillation range for detecting and determining the proportion of kerosine in petrol–kerosine mixtures are discussed. A third method which gives results in close agreement with the first two methods is described. This involves the determination of the b.p. of the motor spirit in an all-glass reflux apparatus, in which the boiling points of mixtures of known composition have been previously determined. J. T.

440. Ring analysis of hydrocarbon mixtures. R. E. Hersh, M. R. Fenske, E. R. Booser, and E. F. Koch. *J. Inst. Petrol.*, 1950, **36**, 624–68.—Methods are developed using only

refractive index and mol. wt. for estimating the average number of rings per mol, the average number of carbon atoms occurring in ring structure, the average wt per cent rings, and the average wt per cent carbon in ring structures for hydrocarbon mixtures consisting of naphthenes and paraffins or of aromatics and paraffins. Application of these methods to 722 pure hydrocarbons (paraffins, naphthenes, and aromatics) shows that the average algebraic deviations between calculated and theoretical values is + 1·5 wt per cent rings, + 0·02 rings per mol, + 0·09 ring carbon atoms per mol, and + 0·06 wt per cent ring carbon atoms. A. R. W. B.

411. Gravimetric analysis of exhaust gas from gas turbine combustion chambers. F. W. Ruegg and C. Halpern. *Bur. Stand. J. Res. Wash.*, 1950, **45** (2), 113.—The gravimetric method of analysis has been applied not to identify all of the constituents of exhaust gas but primarily to determine the efficiency of the combustion process. Carbon dioxide and water are determined by change of weight of solid adsorbents. Combustion efficiency determined by this method agreed to ½% with that calculated from the composition of prepared samples and the combustion efficiency obtained by the analysis of actual gas samples agreed with that calculated by heat balance to better than ½%. J. T.

442. Calorimeter for some corrosive liquids. B. H. Sage and E. W. Hough. *Anal. Chem.*, 1950, **22**, 1304–9.—The calorimeter described is suitable for measuring the heat capacity of corrosive liquids at temperatures up to 400° F. This instrument follows the general design utilized by Osborne and co-workers for the study of the heat capacity of water. The calorimeter appears to be capable of yielding results with an uncertainty of less than 1% provided the rates of corrosion and decomposition of the fluid under investigation are sufficently small. The calorimeter was constructed of stainless steel containing a large amount of chromium and nickel, and was gold-plated on the internal surfaces to decrease further the rate of corrosion by such materials as red fuming nitric acid. Agitation was provided in the calorimeter to ensure thermal and phase equilibrium, and the assembly was confined within an adiabatic vacuum jacket. Provisions were made for the addition electrically of known amounts of energy to the calorimeter and its contents and for measurements of the resulting change in state. From these and other data, the heat capacity of the liquid was established. J. S.

443. Counter-current distribution. G. Kalopissis. *Chim. et Ind.*, 1950, **64**, 563–73.—A study of counter-current liq–liq extraction as applied to laboratory separation and quant determination of complex mixtures of chemically similar constituents, such as members of a homologous series or isomers. Published work (twenty-six references) is reviewed in detail with particular reference to that of Craig (*Anal. Chem.*, 1949, **21**, 500). Apparatus used is described, and the mathematical expression (in binomial form) of the process considered. Applications of the method have, so far, been mainly in the field of biological chemistry. V. B.

444. Methods of knock-detection. R. Vichnievsky and R. Guyot. *Rev. Inst. franç. Pétrole*, 1950, **5**, 371–86.—Various procedures utilized for knock detection are reviewed: electro-acoustic; measurement of cyl-head vibrations; direct determination of pressure vibrations due to knock; ionization methods. A chronological table of these and other methods is given. Tests made at the Claude Bonnier research establishment are described in some detail. Microphone methods were not found suitable, but a procedure based on vibration detection was developed and improved. Readings are shown on a cathode-ray tube, and fuel evaluation can be based on the distance (along the time base) of knock vibrations from T.D.C.; the distance increases with O.N. Experimental work was done on a CFR engine but method can be applied to multi-cyl engines. A liquid potentiometer was developed, with two immersed rotating electrodes coupled to the test engine, which enables vibrations to be recorded on a cathode-ray tube. Circuit diagram is given. V. B.

445. Effects of pressure and other variables on determinations of octane number. W. G. Levedahl. *Bur. Stand. J. Res. Wash.*, 1950, **45** (2), 148.—Knock ratings at altitude differ significantly from those at sea-level by both the A.S.T.M. Motor and

Research Methods. Experiments were carried out in an altitude chamber to determine the effect on O.N. of ambient pressure, engine speed, inlet mixture temp, spark advance, knock intensity, exhaust back pressure, and dia of carburetter venturi. The results show that no changes short of supercharging the engine will give ratings identical with those determined at sea-level. J. T.

446. French mass spectrometer for the petroleum industry. III. Comparative study of methods for the analysis of light hydrocarbons. T. Reis. *Rev. Inst. franç. Pétrole*, 1950, **5**, 387–404.—(cf. Abstract No. 1708—1950.) In order to compare other analytical methods with the mass spectrometer a review is given of published analytical procedures applicable to hydrocarbons having b.p. approx $<100°$ C. Procedures are divided into separation methods (various types of dist; adsorption), chemical and combustion analysis; physical methods (optical and other). Principles of each of the procedures are briefly outlined. It is considered that most scope is afforded by joint use of infrared and mass spectra; there are indications that micro-wave spectrometry may have an important future. 151 references. V. B.

447. X-Ray absorption spectrometry for the determination of T.E.L. in gasoline. Investigation of mono- and polychromatic methods and description of monochromatic method for routine use. H. K. Hughes and F. P. Hochgesang. *Anal. Chem.*, 1950, **22**, 1248–58.—Various X-ray-absorption methods have been investigated experimentally with respect to their sensitivity for the determination of T.E.L., their freedom from interference by sulphur and base gasoline variations, and their practical usefulness. A monochromatic method, using two thorium X-ray lines near a lead-absorption discontinuity, is recommended as a reasonably foolproof, rapid method for routine use. In the absence of commercially available thorium-target X-ray tubes, a monochromatic method employing one molybdenum X-ray line is currently in use. A sample is analysed in less than 7 min with an accuracy that averages better than 0·1 ml of T.E.L/gal. When T.E.L. is blended into a gasoline of known stock, the max error should equal the reproducibility, which is 0·03 ml of T.E.L/gal. The method is compared to chemical and polarographic methods with respect to speed, accuracy, and cost. J. S.

448. X-Ray fluorescence analysis of ethyl fluid in aviation gasoline. L. S. Birks, E. J. Brooks, H. Friedman, and R. M. Roe. *Anal. Chem.*, 1950, **22**, 1258–61.—X-ray fluorescence analysis was applied to the quantitative determination of lead and bromine in aviation gasoline. The characteristic X-ray lines used were the lead L line of wavelength 1·17 Å and the bromine K line at 1·04 Å. A 1-min count at the peak of the lead line gave a probable error of $\pm$ 0·06 ml/gal in a content of 4 ml of T.E.L/gal of gasoline; for the same counting interval, the bromine line could be measured with a probable error of $\pm$ 0·16 ml/gal in 1·8 ml of ethylene bromide per gallon of gasoline. The statistical accuracy improves in proportion to the square root of the length of the counting period. The relative intensities of the bromine and lead lines determined the relative amounts of the two present independently of variations in the base stock. Presence of additives such as chlorine had a negligible effect on the determination of lead and bromine. J. S.

449. Determination of T.E.L. in gasoline by X-ray absorptiometry. G. Calingaret, F. W. Lamb, H. L. Miller, and G. E. Noakes. *Anal. Chem.*, 1950, **22**, 1238–48.—The results obtained in an investigation of X-ray absorptiometry as a method for the determination of T.E.L. in gasoline are described. The sensitivity of the absorbance measurements to voltage fluctuations made it imperative to be able to adjust and maintain the primary voltage constant to $\pm$ 0·10 volt. The refinements necessary to obtain this voltage constancy are given, as well as other modifications made to the General Electric X-ray photometer which resulted in improved performance. The sensitivity of the absorbance measurements of gasoline samples to voltage fluctuations was decreased by a factor of 10 by the introduction of a polystyrene block in the reference beam. The calibration data obtained for T.E.L. in gasoline showed the method to be sensitive to 0·01 ml of T.E.L/gal. The precision of the method is $\pm$ 0·01 ml of T.E.L/gal. In the analysis of samples for which the respective unleaded base stocks are available, the results are accurate to $\pm$ 0·01 ml of T.E.L/gal. The analysis

of sixty-eight samples for which the respective base stocks were not available showed the results to be accurate to ± 0·05 ml of T.E.L/gal. The varying percentage of sulphur in gasoline was found to be the chief obstacle to a more accurate determination of T.E.L. by X-ray absorptiometry. Possible methods for eliminating problems due to varying sulphur and halogen concentrations are discussed. All apparent anomalies encountered in absorbance measurements are accounted for by scattering and filtering effects. J. S.

450. Rapid determination of T.E.L. in aviation gasoline. V. A. Smith, W. E. Delaney, W. J. Tancig, and J. C. Bailie. *Anal. Chem.*, 1950, **22**, 1230–1.—A new and rapid method for the determination of T.E.L. in aviation gasoline is based on the reaction of alcoholic silver nitrate with T.E.L. to form metallic silver. The silver, present as a colloidal suspension, is determined by turbidimetric methods using a photo-electric colorimeter. The method is applicable to the determination of the T.E.L. content of aviation fuels and motor-fuel stocks sweetened with copper chloride, but cannot be used for doctor-sweetened stocks because of interference by free sulphur or polysulphides. Results on aviation fuels check closely with the A.S.T.M. method D526. J. S.

451. A direct-reading polarograph for the determination of T.E.L. in gasoline. E. B. Offutt and L. V. Sorg. *Anal. Chem.*, 1950, **22**, 1234–7.—The successful investigation of antimony as a pilot ion in the polarographic determination of lead has made possible the construction of an instrument by which the lead content of gasoline may be read directly from a calibrated dial while the dropping mercury electrode is polarized in the hydrochloric acid extract from the oil. Predetermined potentials are applied through a sequence of push-button operations. Compensation of the resulting currents is accomplished by adjustment of potentiometers, using a galvanometer as a null instrument; the last such adjustment gives the reading of T.E.L. concentration in ml/gal. Provision has been made for rapid standardization of the instrument and repetition of the push-button sequence for check determinations. Comparisons of this polarographic method with the standard A.S.T.M. method for the determination of T.E.L. in gasoline show that the polarographic method is at least equivalent to the A.S.T.M. method with respect to both accuracy and reproducibility. J. S.

452. Rapid polarographic determination of T.E.L. in gasoline. K. A. Hansen, T. D. Parks, and L. Lykken. *Anal. Chem.*, 1950, **22**, 1232–3.—A rapid, direct, polarographic method for the determination of T.E.L. in gasoline is described. The sample is dissolved and the T.E.L. is decomposed in anhydrous cellosolve containing hydrogen chloride, and the lead ions in the resulting solution are determined directly by the polarograph. The method is applicable to T.E.L. in gasolines in the range of 0·5 to 8 ml per gal, and is generally accurate to within ± 3% of the lead content in freshly prepared blends. Aged gasolines containing high concentrations of unsaturates and peroxides may give appreciable errors. A single determination requires about 30 min, but a series of 5 determinations requires only 1 hr. J. S.

453. Significance of ignition delay and combustion temperature in self-ignition in the diesel engine and in knocking in the petrol engine. W. Witschakowski. *Rev. Inst. franç. Pétrole*, 1950, **5**, 339–54.—Similarity between mechanism of knock in spark-ignition engine and ignition delay (self-ignition) in C.I. engine is discussed, and forms basis for evaluation of spark-ignition-engine fuels by an ignition-delay method. Engine used was a single-cyl German (I.G.k.) diesel test engine. Ten fuels (physical properties listed) were examined, ignition-delay periods (sec 10^{-3}) varied from 0·53 (cetane) to 2·7 (*tert*methyl-butyl-ketone). Ignition delay can be expressed by the Semenoff formula:

$$z = \frac{a \cdot e^{b/T}}{p^n}$$

where z is ignition delay (sec 10^{-3}), T temp (° K), p pressure (kg/cm²), and a, b, and n constants which are characteristic of the fuel. Values of a and b/n are given for the fuels tested. a is affected by additives such as T.E.L., and if a' is value for a fuel containing T.E.L. and a that for a non-doped fuel ratio a'/a expresses the Pb susceptibility of the fuel; b and n are independent of additives. Use of the procedure

described enables a single scale to be employed in place of O.N. and Cetane No., thus for diesel-engine fuels a suitable value of a is 10–15, for automobile fuels about 50, and >280 for the highest grade of aviation fuel. Knowledge of the above constants enables the ignition delays for various engine-operating conditions to be calc. V. B.

454. Interim report of the Oxidation Panel. Anon. *J. Inst. Petrol.*, 1950, **36**, 669–71. —Appendices IV and V inadvertently omitted from this report when it was published in *J. Inst. Petrol.*, 1950, **36**, 336–44. A. R. W. B.

455. Direct determination of oxygen in compounds containing carbon, hydrogen and oxygen. A physical-chemical technique. C. C. Harris, D. M. Smith, and J. Mitchell, Jr. *Anal. Chem.*, 1950, **22**, 1297–304.—A new technique is presented for the direct determination of oxygen in organic compounds, based on the Unterzaucher carbon-reduction procedure. The sample is decomposed in a stream of helium, and the pyrolysis products are circulated in a closed system over carbon at 1100°–1150° C, until conversion of all the oxygen to carbon monoxide is complete. During circulation, essentially all the hydrogen is removed selectively by diffusion through a heated palladium tube. The procedure permits the use of very large samples, which is of particular value in the analysis of materials of low oxygen content. Using a thermal-conductivity bridge, the composition of the resultant helium–carbon monoxide mixture is determined with a sensitivity of 0·20% carbon monoxide per millivolt. The amount of carbon monoxide formed from the oxygen in the sample may be calculated after measurement of the total vol of gas. Because the thermal-conductivity measurements are referred to a control cell, tank helium may be used without further purification. By employing continuous recording of bridge potential, a valuable means is provided for following the course of pyrolysis and conversion. Preliminary results on a wide variety of solid and liquid compounds indicate a relative accuracy of 1% or better. J. S.

456. Microdetermination of sulphur by the Grote method. Photometric detection of the titrimetric end-point. R. N. Walter. *Anal. Chem.*, 1950, **22**, 1332–4.—In the Grote combustion micro-method for determining sulphur in organic compounds, the sulphate formed is usually titrated with standard barium chloride solution, using tetrahydroquinone or an alkali of rhodizonic acid as an internal indicator. A photometric method of detection of end-point, which is more objective than the usual visual method, is described. Titration results are reproducible within 0·004 mg of sulphur. J. S.

457. Reduced-scale methods for micro and semimicro analysis. Part II. T. D. Parks and L. Lykken. *Refiner*, 1950, **29** (9), 112–14.—The paper gives reduced-scale methods for the following inorganic elements: silica, calcium, magnesium, tin, phosphorus, aluminium, sodium and potassium, silver, nickel, copper and iron, lead, and zinc. Detailed procedures are given for silica, calcium, tin, and nickel. Many inorganic samples originate from motor tests or corrosion studies when only small amounts are available. In these cases a preliminary spectrographic examination serves to indicate the best method of analysis by showing major constituents present. The article contains twenty-seven references to the literature. R. W. G.

458. Glycol ethers as non-aqueous solvents in polarographic analysis. T. D. Parks and K. A. Hansen. *Anal. Chem.*, 1950, **22**, 1268–9.—The ethylene glycol mono-alkyl ethers (Cellosolves) are satisfactory non-aqueous solvents for the polarographic study of organic compounds. They are chemically and polarographically stable, dissolve certain polar materials to give conducting solutions, and are miscible with a wide variety of materials, including water and gasoline. Many non-polar organic compounds as well as metal ions give satisfactory polarograms in these non-aqueous media. J. S.

459. Flame tests in the presence of sodium. R. B. Hahn. *Chem. Education*, Nov. 1950, **27** (11), 597.—The method of using the blue cobalt glass filter to make flame tests in the presence of sodium is not entirely satisfactory, since part of the spectra

emitted by elements other than sodium is also absorbed by the filter, which alters the characteristic colour and decreases the sensitivity of the test. A didymium glass filter serves the purpose much better than the cobalt glass. It transmits the light unchanged in colour and practically undiminished in intensity. A study of the limit of sensitivity of various flame tests was made, and the results of this are given. A. S.

460. Methods of testing adhesion of paints. R. Quarendon. *Paint Mnfr.*, Dec. 1950, **20** (12), 427.—Various tests devised or suggested for the measurement of " adhesion " of a paint film to the base metal are discussed. The limitations of the tests and an account of the latest developments in the measurement of adhesion are included. The tests are roughly classified into tensile, deformation, scratching or scraping, impact, and direct adhesion. Specific tests discussed in more detail are : the Courtney and Wakefield test, Schmidt tensile test, the use of the Gardner and Parkes tensile strength meter, the Gelva test, the mandrel or bending test, and Ministry of Supply impact tests. The American methods include the New York Production Club Method and the use of the Bell Telephone Laboratory distensibility machine. Apparatus described includes the Erichsen tester, impact testers, rate-of-impact testers, and the vibrational adhesion tester. The relative merits of the various methods are considered. It is believed that the Moses and Witt technique, followed in the Courtney and Wakefield method and the Schmidt test, is the most satisfactory for a fairly scientific measure of adhesion itself. The mandrel and impact tests give results which depend to a considerable extent on the properties of the film and can be regarded as measuring a composite of adhesion, elasticity, cohesion, and other properties. Several tests may be used in an investigation into adhesion, and are generally employed to compare the behaviour of different paint films under given conditions or of one film under different conditions. There are fifteen literature references. D. K.

461. The viscosity of linseed oil lithographic varnish films during oxidation drying. W. H. Banks and P. B. G. Upton. *J. Oil Col. Chem. Assoc.*, Sept. 1950, **33** (363), 397.—A method is described of measuring the visc of a thin film of linseed oil. A sheet of glass 100 × 30 cm was supported so that it could be tilted at known angles and in such a manner that it was in one plane and suffered no sagging. A known vol of varnish was uniformly distributed over the entire plate. The thickness of the resulting film was calculated from the vol supplied by assuming the varnish to be equally distributed. A steel bearing was allowed to roll down the inclined glass plane and the speed of roll measured. The method was calibrated with oils of known visc. The influence of film thickness and angle of tilt was studied. Measurements were made of the time taken for the ball to roll between points 60 cm apart and for different angles of tilt. Measurements were made on varnishes during oxidation. The variation in mol. wt. during oxidation and the relationship between mol. wt. and visc are also discussed. All results are graphed or tabulated. D. K.

462. On the properties of water surface in contact with various vapours. T. Ikeda and T. Ozaki. *Bull. Chem. Soc. Japan*, July 1950, **23** (2), 43.—The apparatus and procedure used for determining the surface tension of water in various vapours by the drop-volume method are described, and the results obtained with benzene, *n*-hexane, carbon disulphide and several chlorinated compounds, alcohols, and ethers are recorded. The " ageing " of the water surface and its mechanism, the vapour-water interfacial tension and the relation to 'Antonoff's rule, and effects of pressure are discussed. It was found that the 'Antonoff's rule holds good with some exceptions. In any vapours a remarkable, but short, ageing of water surface was found, which was 6 sec in general, and which is prolonged by reduced pressure. W. H. C.

Crude Oil

463. How to improve A.P.I. Code 25. G. H. Supple and C. Nicholson. *Oil Gas J.*, 16.11.50, **49** (28), 346.—Comments based on a comparison of reported current practices of certain companies with A.P.I. Code 25 concerning measuring, sampling, and testing crude oil are given. A new committee on A.P.I. Code 25 has been formed. G. A. C.

Gas Oil and Fuel Oil

464. Effect of fuel viscosity on injection equipment. J. G. Withers. *Gas Oil Pwr*, 1950, **45**, 146–8.—The effect of fuel visc on leakage past the working surfaces in fuel-pumps and injectors is examined.

The visc of diesel fuel oil may vary from 1·4 to 7·5 cs at 100° F, and larger variations are encountered in practice due to a range of temp from 32° to 212° F. The London Transport Executive has developed a test in which a predetermined pressure drop through the pump or injector is timed. For a pump this time must exceed 3 sec over a pressure drop from 250 to 150 atm; for an injector the time must exceed 8 sec for a pressure drop from 95 to 70 atm.

It can be shown that, even for a badly-worn injector or injection equipment, the fuel leakage obeys the laws of streamline flow. This has been confirmed by plotting the % of injection leakage against 1/absolute visc, when a straight-line relationship is found. It is concluded that under the worst conditions leakage will be $< 1\%$ of the fuel delivered.

New and worn fuel-pumps were compared under standard conditions, and it was found that there was a difference of 4% between the max outputs of the units. For fuel-pumps the % leakage *versus* 1/absolute visc graph is not a straight line, since the larger pressure drop through delivery valve and connecting pipe with high-visc fuels causes a relatively higher pressure than with low-visc fuels.

There is no evidence that reduction in fuel visc causes higher rates of wear. Low-visc fuels give a finer spray with shorter penetration into the combustion space.

H. C. E.

Lubricants

465. Some interesting points on lubrication. H. Nicol. *J. Inst. Mar. Engrs*, Nov. 1950, **72** (11), 342.—The purpose of this paper is to describe for the benefit of junior marine operating engineers the amount of time and thought which has to be given to the intricacies of efficient lubrication and the fundamental principles involved in the lubrication of the most important parts of a diesel engine. Elementary information is first given on the general properties of lubricants and the phenomenon of friction and wear. The lubrication of diesel engines is then dealt with, and particular reference is made to systems of forced lubrication, designs of main, crankpin and crosshead pin bearings, the Michell thrust block, mechanical sight-feed cyl lubricators. A large number of diagrams and drawings are included in the paper. A. S.

466. Lubrication life forecast and test procedure. H. D. Martin. *Institute Spokesman*, Oct. 1950, **14** (7), 10.—Details are presented of the methods of grease testing developed for New Departure integrally sealed and shielded ball bearings. The construction and operation of a special testing machine is described, and methods of forecasting and testing bearing life under given conditions are outlined and the results compared. Grease life as affected by type of bearing is indicated, and the effect of outer ring rotation is considered. J. G. H.

467. Blending techniques. E. W. Steinitz. *Petroleum*, Aug. 1949, **12** (8), 194–6.—Owing to growing demand, asphalt-base crudes had to be used for making lubricating oil. Drawbacks of such solvent-refined oils became apparent, however, as requirements of lubricating oil grew more stringent. A classified survey of additives used to overcome the basic defects of such asphaltic crudes is given. These include oiliness improvers, extreme-pressure additives, detergents, V.I. improvers, pour-point depressants, oxidation and corrosion inhibitors. Problems of boundary lubrication, piston lubrication, and water splash are discussed in connexion with additives.

It is suggested that more information should be given by oil manufacturers, *e.g.*, origin and treatment of crude. This would facilitate the work of the oil blender, as would more information on the composition of additives. R. E. P.

468. "Detergency" in crankcase oils. F. F. Musgrave. *Scientific Lubrication*, Oct. 1950, **2** (10), 10–14.—The development, composition, and classification of detergent crankcase lubricating oils are briefly summarized, and the characteristics of

Supplement 2, Supplement 1, Military 2104, and U.S. Navy Series 9000 grades are enumerated. Engine and field tests are described, and the advantages of detergent oils set out, together with their limitations. Other points discussed include the effect of fuel variation and wet sludge. J. G. H.

469. Viscosity–temperature characteristics of lubricating oils. M. M. Kusakov. *Petroleum*, July 1949, **12** (7), 170–4. (*Translated from Symposium on the Viscosity of Liquids and Colloidal Solutions, Academy of Sciences of the U.S.S.R.*)—In the characterization of oil quality, the visc and the variation of the temp gradient of visc with temperature are of great significance. The author points out the disadvantages of the numerous existing systems of V.I. and shows that two or three parameters are necessary to characterize the " level " of visc and the degree of flatness of the visc–temp curves for oils. One parameter denotes the visc at a given temp, and the second, by itself or together with the third, indicates the slope of the curve

$$\eta = f(t).$$

Three constants are sufficient to express the variation of visc with temp, and give sufficient accuracy for the solution of problems involving interpolation and extrapolation, as well as in the accurate classification of oils.

It is proposed to utilize the Vogel–Fulcher–Tammann formula for variation of visc with temp. The three constants of this formula have a precise physical meaning, and enable both the visc level and slope of the visc–temp curve to be conveniently defined. It is shown how to evaluate the constants from a knowledge of the kinematic visc of the oil at three temperatures. R. E. P.

470. Lubrication at extreme temperatures. A. H. Stuart. *Petroleum*, June 1949, **12** (6), 139–41.—Advances in engineering practice have called for lubrication at temp at both ends of the scale. Oils and greases are unsuitable at very low and very high temp due to excessively high or low visc, and also to chemical instability of the oil. The nature of the problem is considered and examples and methods are given of overcoming the difficulties involved.

The advantages of dry film lubrication are discussed with special reference to the properties of graphite. Graphite films may be imposed on bearings by brushing, spraying, or used as a dispersion in water or oil. The advantages of such films are :—

(1) lubricating properties unaffected by changes in temperature over a very wide range ;

(3) substantial reduction in coefficient of friction, and motion between members is smooth and regular—does not show " stick-slip " phenomenon as do lubricating oils ;

(3) acts as a parting compound, preventing adhesion of metals ;

(4) encourages oil spread if mixed with normal lubricating oil ;

The life of such films is also considered. R. E. P.

471. Better lubrication for diesels. B. E. Eltham. *Mech. World*, 1.12.50, **128** (3333), 517.—The lubricant in a diesel engine is subjected to severe oxidation and also contamination, and these severe conditions occur more with high-speed than with slow-speed engines, because in the former, due to higher piston temperatures, there is a greater tendency for piston-ring sticking to take place. Cyl walls, piston surfaces, and rings are the most sensitive engine parts with regard to the kind of oil used. At the temp of the top piston-rings the visc of the oil should be sufficient to maintain effective film lubrication, otherwise increased wear will result. High-visc index oils are therefore required for the prevention of such wear. As far as scuffing is concerned, oiliness is the basic lubricating property of interest. The high temp at the upper piston-ring zone tend to cause " cracking " of the oil, and this results in the formation of resinous asphalts and carbon. These, together with products of combustion, cause sluggish ring action and eventual gumming. In recent years special additives of a detergent nature have been added to the straight distilled oils to increase their oxidation resistance, V.I., and to decrease deposit formation. These detergent additives are also effective in the prevention of sludge deposits. Copper–lead and cadmium–nickel alloy bearings are often used in high-speed diesels because of their ability to

withstand cyl pressures and because of their superior resistance to wear. An oil must be non-corrosive, for these bearings are attacked by acidic compounds formed by oxidizing conditions in the crankcase. A. S.

472. Steam turbine lubrication. Part II. W. E. J. Broom and G. H. Clark. *I.P. Review*, 1950, **4**, 325.—Since modern turbine oils are rarely submitted to pressures greater than 150 p.s.i., their quality in this respect is not difficult to obtain. Other factors, however, are important. (1) There is a min visc (about 40 sec Red. I at the operating temp) below which it is impossible to build a full fluid film. (2) The oil must not cause too much heat loss. (3) The oil must have a high V.I. (4) The oil must not oxidize excessively, which depends on temp, presence of air, and catalytic effect of fine metallic particles in suspension. Attempts were made to improve the quality of turbine oils by solvent extraction; now, the use of additives is widely practised. Corrosion is also a problem; it is alleviated by ensuring effective wetting by addition of a rust inhibitor. This does not prevent corrosion above the oil-level, however. Performance of turbine oils is often checked by noting any change in acidity, but change in saponification value (which is related to the interfacial tension) is more discerning. Methods of turbine-oil maintenance are described. R. H.

473. Centralized lubrication at a steel rolling mill. Anon. *Scientific Lubrication*, Oct. 1950, **2** (10), 15–18.—A description of the Tecalemit "Multipoint" installation at the rolling mills of Partridge Jones and John Paton Ltd. at Pontnewynydd. J. G. H.

474. Story of a new grease. G. S. Bright. *Institute Spokesman*, Sept. 1950, **14** (6), 11–16.—The steps in the development of a new grease from the drawing-board stage of the equipment to be lubricated are outlined. The properties and functions of fatty materials, soaps, oils, and additives are discussed, together with laboratory research and commercial production. J. G. H.

475. Basic factors determining the structure and rheology of lubricating greases. A. Bondi, A. M. Gravath, R. J. Moore, and W. H. Peterson. *Institute Spokesman*, May 1950, **13** (12), 12–18.—The necessity for a clear understanding of the physical properties of lubricating greases as an essential preliminary to further development is stressed, and the variations in soap structure in grease with rise of temp are discussed. Visc, consistency, and grease breakdown are considered in some detail with the Shell Roll Tests and the AXS 1574 Army Wheelbearing Test. J. G. H.

Special Hydrocarbon Products

476. Investment-waxes. Anon. *Aircr. Prod.*, Nov. 1950, **12** (145), 313.—A description is given of a pneumatically-operated injection machine manufactured by Spares and Equipment Ltd., Bristol, for the production of wax-patterns for investment castings. This machine enables the steel die, in which the wax is cast, to be completely and rapidly filled with bubble-free wax, and the variables of temp and pressure can be controlled within close limits. U. N.

477. Waxes. C. J. Marsel. *Chem. Ind.*, Oct. 1950, **67** (4), 563.—Recent industrial activity has resulted in a tremendous growth in the variety of available waxes. This article, in reviewing current wax technology, deals with waxes under the headings plant waxes, animal and insect waxes, petroleum waxes, and earth waxes, and, in most cases, describes their origin, their composition, and the methods by which they are refined. A section is devoted to synthetic waxes such as the stearic esters of polyhydric alcohols and derivatives of ethylene oxide. Condensation products of chlorinated hydrocarbons and naphthalene are specially mentioned in view of their fire-retarding ability.

By judicious blending of various waxes, it is possible to produce a range of waxes fulfilling widely different requirements as regards physical and chemical properties.

A very useful adjunct to the article is a table listing American manufacturers and suppliers of synthetic, blended, mineral, and petroleum waxes, along with chemical and physical properties, principle uses, and special characteristics of the products supplied. O. M.

Derived Chemical Products

478. Chemicals from petroleum. Anon. *Chem. Prod.*, Nov. 1950, **13** (12), 440.—A specially contributed article dealing with the wide range of chemical products which can be produced from petroleum hydrocarbons, with special emphasis on the applications of those which are being, or will be, produced in the U.K. O. M.

479. Relationship between detergency and adsorption by the fibre. K. McLaren. *J. Soc. Dyers Colourists*, 1950, **66** (10), 521.—A general introductory outline to the subject is given, together with a description of the observations which led to the experimental work described. This latter—involving the investigation of the effect of four variable factors, temp and concentrations of detergent, acid and salt on the wool-detergent properties of a specific, non-ionic, surface-active compound—is given in detail. The effect of each factor on the adsorption of detergent by the fibre is also described. C. N. T.

Coal, Shale, and Peat

480. Mode of action of specific solvents for bituminous coals. I. G. C. Dryden. *Nature*, 1950, **166**, 606.—When low-rank coals are extracted with specific solvents (see Abstract No. 481) it is found that :—

(1) Mechanical shaking increases the rate of solution but decreases the yield of soluble material obtained in a single treatment.

(2) The max yield depends only upon the type of coal, the solvent, and the temp.

(3) For the lowest rank coals and the best solvents the concentration of dissolved material is the same both in the solvent imbibed in the coal and in the bulk solvent for dilute solutions. For concentrated solutions the dissolved material in the bulk solvent exceeds that in the solvent in close contact with the coal.

It appears that if the volume imbibition ratio is small, the yield of extract is controlled by the difficulty of dispersion in the solvent; if the volume imbibition ratio is large, the yield is determined by the size distribution of colloidal units.

The temp coefficient of extraction yield varies to a limited extent with the rank of the coal; when the volume imbibition ratio > 1 its value ceases to change further with rank. (See also Abstracts Nos. 630, 866, 1949.) H. C. E.

481. Nature of specific solvents for bituminous coals. I. G. C. Dryden. *Nature*, 1950, **166**, 561.—The ability of more than 100 substances to dissolve low-rank bituminous coals at room temp has disclosed that almost all liquids fall definitely into one of the categories : solvent or non-solvent. It is found that :—

(1) The solvent contains an N or O atom with an unshared pair of electrons which, however, may be prevented from promoting solvent power : (i) by excessive hydrogen bonding (*e.g.*, dilution with water); or (ii) by participation with resonance in the nucleus (*e.g.*, as in pyrrole as contrasted with pyridine).

(2) Other things being equal, N compounds are better solvents than O compounds.

(3) Acetyl groups tend to reduce solvent power.

(4) Solvent power is further modified by molecular size and viscosity.

That an electron-donor mechanism is involved in the interaction between coals and amines is confirmed by determining heats of reaction of amines with $AlCl_3$ as a measure of co-ordinate bond energy; in both cases various amines are ranked in the same order. Observations with amines and Bu_3B indicate that the electron-acceptor on coal surfaces is surrounded by alkyl or other groups projecting from the surface.

H. C. E.

482. Shale retorting process verified by test run. Anon. *Refiner*, 1950, **29** (9), 203.—A short description of the Union Oil Co. of California, Los Angeles, 50-ton/day continuous shale-retorting plant now in operation. Increasing consumption of crude oil reserves and increasing costs for exploration and production of crude oil indicate shale-oil mining and recovery to be commercially sound. R. W. G.

483. Oil shale in Spain. H. M. Thorn and A. J. Kraemer. *U.S. Bur. Mines Rep Invest*, 4736, Oct. 1950.—Brief notes are given of the activities of the Empresa Nacional "Calvo-Sotelo" de Combustibles y Lubricantes, and the oil-shale beds at Puertollano are described and shown in a plan. The characteristics of the Puerto oil shales and yields on retorting are given. The Pennarroya oil-shale plant is described, and the refining and properties of the crude oil obtained, and of the products, are recorded. Research and development are outlined. A bibliography of Spanish literature on shale oil is included. W. H. C.

484. Further investigation of the chemical constitution of the kerogen of oil shales. T. E. Dancy and V. Giedroyc. *J. Inst. Petrol.*, 1950, **36**, 607–23.—The constitution of the kerogen of the Ermelo, St Hilaire, and Kimmeridge oil shales was studied using oxidation methods. The mechanisms of gaseous oxygen and hydrogen peroxide oxidations were investigated, and the oxidation products were compared with those obtained from alkaline permanganate oxidations. Molecular and equivalent wt determinations were made on some of the more complex products, and infra-red spectrum analysis was also used. A. R. W. B.

485. Further researches on the determination of the chemical composition of oil shales. T. E. Dancy and V. Giedroyc. *J. Inst. Petrol.*, 1950, **36**, 593–603.—The removal of mineral matter from the Ermelo Torbanite (South Africa), St Hilaire oil shale (France), and Kimmeridge oil shale (Dorset) was studied using a chemical and a physical method. The former, using mineral acids, caused some attack of the kerogen, and this effect was studied in some detail. The physical method, employing a grinding process with oil, was unsuitable for the Kimmeridge, but was used for the indirect determination of the composition of the kerogen of the other samples. A. R. W. B.

Miscellaneous Products

486. Flake form nonionic detergents. C. R. Pacifico, L. G. Lunsted, and T. H Vaughan. *Soap Sanitary Chem.*, Oct. 1950, **26** (10), 40.—The wide variety of specia properties which can be achieved by modification of the hydrophobic and hydrophilic parts of the molecules which make up non-ionic detergents has been the major reason for the rapidly won popularity of these products and increased off-take has reduced their price.

The one aspect in which "non-ionics" failed to meet demand was the manufacturers inability to make from them flaked products, due to the fact that, if "non-ionics" have the correct balance of hydrophobic and hydrophilic properties to give good detergency, they are almost invariably liquids. This problem has now been solved by employing in the molecule a hitherto unused hydrophobic unit which allows high-mol.-wt. products, with the required degree of detergency, to be manufactured. Such a product can be flaked commercially. This range of products, including liquids, pastes, and solids, is identified by the name "Pluronic."

By means of tables and curves, this paper compares these "Pluronics" with two commercially available non-ionic detergents from such important points of view as foam height, wetting power, carbon soil removal, and hygroscopicity. Since these products have not yet been released to the market, no information regarding their composition is disclosed. O. M.

487. Synergists widen scope of "non-toxic" insecticides. M. Goldberg. *Chem. Ind.*, Oct. 1950, **67** (4), 578.—Even though they are highly efficient, the application of synthetic insecticides, such as D.D.T., is limited by reason of resultant breeding of resistant insects and their toxicity to warm-blooded animals. Therefore, pyrethrum, which does not suffer from these drawbacks, is still an important insecticide.

To increase the activity of pyrethrum has been the aim of research over a long period of time, and this work, through the discovery of the desirable effects connected with the methylene dioxy-phenyl group present in sesame oil, has led to the development of synergists, the best-known of which is piperonyl butoxide. Pyrethrum, synergized with piperonyl butoxide, being non-toxic to mammals, easy to formulate, and rapid in action, has now been widely adopted for garden sprays, cattle dips, household aerosols,

etc. The fact that these formulations, marketed as Pyrenones, can be made up as emulsible concentrates is an obvious advantage over older pyrethrum insecticides.

Other synergists used are sesamin, *n*-propyl isome, and Sulfoxcide. The latter two of these are of particular interest in view of their marked synergistic effect when used with allethrin. This interest also applies to *n*-octyl bi*cyclo*heptene dicarboxinide or " 264," which is particularly potent against cockroaches.

As it is unlikely that allethrin will wholly replace pyrethrum, future development in synergists will probably be directed towards expanding the utility of pyrethrum and enhancing the effectiveness of allethrin. Also synthesis of other pyrethrum-like materials will produce a demand for new synergists. O. M.

488. Oil absorption of pigments and extenders. G. E. Bessey and K. A. Lammiman. *J. Oil Col. Chem. Assoc.*, Sept. 1950, **33** (363), 411.—Recent work carried out in the laboratories of the Research Council of the British Whiting Federation, involving examination of bulk densities of compacted powders and powder–oil mixes, has shown conclusively that all the voids in a powder–oil mixture are completely filled at the oil-absorption E.P. The true density of any mixture of the oil and powder can be calculated, assuming no chemical reaction, as an additive function of the densities of the powder and the oil. A graph is given in which density of mix (g/ml) is plotted against vol of oil per 100 g whiting (ml). Mixtures with insufficient oil to fill all the voids and mixtures with higher oil contents are discussed. If the time of mixing is inadequate, a lower density for any particular mix and a higher value for the oil absorption are obtained. There are four literature references. D. K.

CORROSION

489. Anti-corrosive compositions. Results of B.I.S.R.A. research on underwater service on steel. Anon. *Paint Mnfr*, Dec. 1950, **20** (12), 439.—Sixty-eight anti-corrosive and one anti-fouling composition were prepared and applied to mild-steel specimens in standardized surface conditions. The painted specimens were tested either by immersion in sea-water from rafts or by the C.R.L. accelerated rotor test. For the raft tests three coatings were applied two of anti-corrosive compositions followed by one of anti-fouling composition. For the rotor tests just two coats of anti-corrosive were used. The compositions used comprised variations of mixtures of basic lead sulphate, white lead, aluminium powder, Burntisland red, and barytes. All pigmentations were duplicated in compositions bound respectively with a modified phenol-formaldehyde/stand-oil medium and a coumarone/stand-oil medium. Three of the pigmentations were bound with each of five other media, including chlorinated rubber, and media derived from modified phenol-formaldehyde/stand-oil by the substitution of dehydrated castor oil or tung oil for part of the stand-oil.

The best performances were given by paints bound in chlorinated rubber. These were closely followed by a phenol-formaldehyde/stand-oil/tung-oil medium. The effect of pigmentation was slight. Increase in aluminium tended to give improved performance as regards protective properties and performance. The most promising composition appeared to be 2 parts basic lead sulphate with 1 part each of aluminium powder, barytes, and Burntisland red bound in a modified phenol-formaldehyde/stand-oil/tung-oil (1 : 1) medium. Two tables are included, one giving the main results of the raft and rotor tests, and the other a summary of the formulations and physical properties of the compositions. D. K.

490. New corrosion inhibitor gets job done. K. T. Feldman and F. M. Watkins. *Oil Gas J.*, 16.11.50, **49** (28), 340.—Four years' experience with rust inhibitor RD-119, a stable product soluble in distillate fuels developed by the Sinclair Refining Co., U.S.A., and used to prevent corrosion in the interior of products pipelines is reported.

The mechanism of the inhibitor action lies in polar molecules adsorbing on metallic and metallic-oxide surfaces, thereby decreasing tendency of water and of corrosive water solution to wet and corrode.

Inspection data on test coupons showing corrosion rating after exposure on two pipeline systems are given.

Maintenance of equipment coming into direct contact with product streams has been much easier since RD-119 has been used as a rust inhibitor. G. A. C.

491. Corrosion, derusting, inhibition and passivity of metals. (Article in German.) J. Frasch. *Ingenieur*, 20.10.50, **62** (42), Mk. 87.—A study is made of the means available of combating the corrosion of iron and steels. Data concerning the nature, properties, and changes of the iron oxides formed during the corrosion process are given. An electrolytic study is made of the parts played by oxygen and hydrogen in the formation of passivity films and the later destruction of these films. Oxygen, the originator of corrosion of iron, at the same time inhibits the corrosion process by forming protective oxide films.

Mechanical and chemical methods of de-rusting are discussed. De-rusting by other reduction processes and de-rusting with hydrochloric or sulphuric acids are briefly reviewed. Phosphoric acid treatment by cold- and hot-dipping, brushing, spraying, etc., is examined in detail. Phosphoric acid solution de-rusting is considered particularly advantageous because of its effect in increasing surface passivity.

Distinctions are made between inhibiting corrosion, protecting surfaces against corrosion, and rendering surfaces inactive for short periods of time. Inhibition and the inhibiting power of certain substances are examined from an electro-chemical viewpoint. Anodic and cathodic inhibition are examined and compared.

Details are given on processes for the formation of passivity films, special mention being made of the gelatine-chromate solution process. Several tables and diagrams are included. L. W.

492. New developments in coatings and cathodic protection. C. L. Goodwin. *Oil Gas J.*, 16.11.50, **49** (28), 355.—Coal-tar enamels, reclaimed synthetic rubber, and an enamel produced by reacting asphalt and resinous material are among the developments in pipeline protection which are discussed.

A review is made of improvements in rectifiers used in cathodic protection methods, the use of carbon or graphite anodes and improvements in galvanic anode installation, ground beds for lighting protection, and other corrosion-control measures.

G. A. C.

493. Metals for high-pressure hydrogenation plants. G. A. Nelson. *Refiner*, 1950, **29** (9), 104–10.—A review is made of suitable materials of construction for handling hydrogen at high pressures and elevated temp. Although hydrogen is an easy material to handle at ordinary pressures, at high pressures it may cause failure of steel. The hydrogen permeates the steel and produces methane by hydrogenation of the iron carbide. This methane, being unable to diffuse out of the metal, leads to cracked grain boundaries and resultant weakness. The attack by hydrogen on steel is greatly reduced by additions of carbide-stabilizing elements. All the austenitic steels are resistant because of their high chromium content. The addition of non-carbide-forming elements (*e.g.*, nickel and silicon) have no effect. A temperature–pressure plot for satisfactory working conditions for carbon and alloy steels is given. For extremely high pressures, steels would be free from hydrogen damage when the alloy to carbon contents were as :—

Ti to C = 4 to 1,
V to C = 5·7 to 1,
Mo to C = between 30 to 1 and 60 to 1.

When other gases, such as H_2S, N_2, and CO are present, further provision must be made. For complete sulphide resistance the high-alloyed steels, such as 12% Cr or 18% Cr–8% Ni, are required. Nitrogen may cause nitriding, but as this does not normally affect more than a $\frac{1}{32}$-inch-deep layer, it is not so troublesome. Carbon monoxide at 260° C is particularly corrosive to steel at 250 and 700 atm. A suitable material of construction is diffusion-galvanized 5% Cr steel. R. W. G.

494. Insulation of pipe fittings with plastics. J. A. Clay. *World Oil*, Apr. 1949, **128** (13), 157.—The effects of stray electric currents contributing to corrosion have long been overlooked. The effects of these currents, if distributed evenly over the equipment, would be inconsiderable, but excessive local corrosion indicates that they are, in fact, concentrated. Chemical corrosion also is aggravated by local currents. Consequently, the principle of breaking the circuit was introduced, and the use of

plastic insulators proposed. A laminated, phenolic-resin, fabric-based plastic was selected.

The practical application of this is in the insulation of pipes connected by flanges. Data are given to assist in the design of the insulators. A. J. H.

495. Internal pipe coating techniques. J. K. Alfred. *Oil Gas J.*, 16.11.50, **49** (28), 266.—Three methods of combating internal corrosion of pipelines, including plastic lining in place, cement lining applied in the mill or local plant, and cement lining applied in place are dealt with.

Plastic lining in place method involves use of two double-opposed rubber plugs for the purpose of containing the cleaning medium or the plastic coating in the pipe; the plugs being forced through the pipeline at controlled speeds by compressed air or inert gas. Thus pipe walls are cleaned by friction and coated by wiping action.

Materials used for cement lining in the mill are essentially mixtures of first-grade hydraulic cement, limestone slag, and native West Texas blow sand, a predetermined quantity of wet mixture being loaded into each length of pipe, the pipe spun for 1¼ min and then uncapped and drained.

Cement lining in place is accomplished by winching a charge of cement and a coating device through the pipe.

The advantages and disadvantages of each method are given, and four figures illustrate the article. G. A. C.

496. Selection and maintenance of tanks in sulphide areas. H. D. Murray. *World Oil*, Apr. 1949, **128** (13), 161.—Corrosion problems in sulphide areas are frequently increased by the production of salt water. Factors governing the selection of suitable tanks include the length of lease and salvage value of the tanks at the end of the lease, corrosion characteristics and the possibility of protection by plastics or by galvanizing, and the variation in production rates. The saleable value of the lighter hydrocarbons has encouraged the reduction of evaporation losses.

The design of tanks should at all times take into account the flexibility of operation due to variation in both oil and water production, and should allow for current improvements. The use of cone-bottom tanks had been restricted by constructional difficulties, but recent introduction of these has increased; the easy removal of sediment and corrosive fluids reduces internal corrosion. Welded tanks are advantageous, since they eliminate the need for gaskets, as compared with bolted tanks.

A table gives the relative merits of wooden, bolted, and shop-welded tanks.

A. J. H.

Engines and Automotive Equipment

497. Lower unit fuel consumption a national need. A. Taub. *Auto Ind.*, 1.3.50, **102** (5), 30–5, 58–63.—The need for lowering unit fuel consumption in internal-combustion engines is emphasized from both military and production angles. Major savings must come from part throttle operating, which is considered from the standpoints of higher C.R., improved thermal efficiency, burning of leaner mixtures, and higher operating temp. Full-throttle fuel improvement is treated from the angles of highest useful C.R., thermal efficiency, internal cooling, detonation, and shock control. The value of F head engines in combination with a partial stratified mixture as an intermediate step is stressed. It is concluded that the final solution to the problem is the single-valve engine. J. G. H.

498. No harmful pre-ignition from lead deposits. A. Hundere. *Auto Ind.*, 1.9.50, **103** (5), 50, 76–83.—Results are presented of tests conducted in a full-scale single-cyl engine to study the pre-ignition characteristics of combustion-chamber deposits. Details of the tests are outlined, and the following conclusions recorded. Lead deposits are incapable of supporting pre-ignition of sufficient advance to have any harmful effect; certain compounded oils contain materials that on burning in the combustion chamber leave deposits capable of supporting destructive pre-ignition; any fuel or oil constituents that on combustion leave deposits having a melting point of about 2000° F can cause incipient pre-ignition, and if the melting point is above about 2500° F destructive pre-ignition can result. Metals present in the oil from normal engine wear

are insufficient in quantity to cause harmful pre-ignition; there is much scope for alleviating lead fouling of sparking plugs without reducing the lead content of the fuel or developing an improved scavenger. J. G. H.

499. Fuel injector of new design. Anon. *Gas Oil Pwr*, 1950, **45**, 184.—The Aphonic injector is claimed to reduce diesel knock, giving even and quiet combustion without increase in fuel consumption. It operates at 100 atm injection pressure and gives a spray angle of 140°. The type of spray is claimed to reduce the rapidity of pressure rise when combustion begins, controls the flame rate, and reduces both engine noise and shock loading. H. C. E.

500. Carburation and induction systems. Anon. *Auto Engr Annual Extra No.*, 30.11.50, **40** (534), 388.—The paper contains descriptions of various types of carburation and induction systems encountered at the 1950 Motor Show at Earl's Court. The conclusions reached are that although a number of new engines were exhibited there was little departure from conventional design in regard to the above two features. Petrol injection appears to have made no progress, and carburettor design indicates no radical changes, although in America the side-entry down-draught seems to be gaining some ground. Cold-starting systems are fairly evenly divided between the off-set strangler valve with linked fast idle mechanism and the auxiliary starting carburetter. Fully automatic cold-starting systems are an almost universal fitting on American cars, although not so general in this country. Regarding induction systems, there is still a good deal of variety in the design and construction of British types, although American vehicles appear to have settled down to the down-draught carburetter using automatic choke, feeding a cast-iron induction system with the exhaust manifolds on the same side. The most popular arrangement on British vehicles is a down-draught open-choke carburetter feeding an induction system located on the same side as the exhaust manifold with a hot spot of some kind between the two.

Descriptions are given of two-, four-, six-, and eight-cylinder engines including British, European, and American types. A. S.

501. Desirable improvements in oil engines. Anon. *Mech. World*, 10.11.50, **128** (3330), 456.—Too many classes of engines are seeking to operate on too narrow a band in the distillates of crude petroleum. The jet engine and gas turbine have brought a tremendous demand for the same range of oil as is used in the heavy-road-vehicle industry and in rail traction. In wartime the demand for this type of fuel for aircraft would obviously have priority over road and rail requirements. Even in peacetime increasing demands for this fuel may cause a serious difficulty unless the gas turbine can be made to run on heavier fuels or pulverized solid fuels. Gas turbines are handicapped by high fuel consumption, and the only way in which they can be a commercial success is by operating on cheaper fuel than that used in oil-engines. Alternatively, bi-fuel systems may be a solution. High-speed diesels still require development to eliminate the disdavantage of noise, smoke, and inflexibility. For rail traction mechanical drive is satisfactory up to 350 b.h.p., but for higher h.p.s the drives are in the experimental stage. Mechanical drives are badly needed to bring the capital cost of the diesel locomotive nearer to that of corresponding steam locomotive. In marine applications the wider acceptance by shipowners of the variable-pitch propeller would often improve the use of diesel engines, and might be the key to the adoption of gas turbines for ships. A. S.

502. Aircooled two-stroke diesel engine of unique design. W. E. Meyer. *Auto Ind.*, 1.5.50, **102** (9), 34–6.—Details are presented of an air-cooled high-speed, two-stroke-cycle diesel engine developed by the Austrian Saurer Corporation for use in tanks and armoured vehicles. War conditions precluded the building of a complete engine, but all necessary single-cyl tests had been concluded, and the design for the full-scale engine was completed. Principle specification points were: twelve cyl in 90° V-arrangement; bore 4·34 in; stroke 6·30 in; displacement 92·7 cu. in. cyl, 1107 cu. in. total; distance between cyls in same row 6·61 in; compression ratio 13·5 : 1; total weight; 3500 lb with cast-iron crankcase; output—max 700 h.p. at 3000 r.p.m., continuous 600 h.p. at 2600 r.p.m. The most noteworthy feature of the engine, its scavenging system, a combination of uniflow and mushroom scavenging is described in detail, and performance curves for the full-scale engine are included. J. G. H.

503. Combustion performance of a swirl chamber diesel engine. A. L. Wachal and J. G. Withers. *Gas Oil Pwr*, 1950, **45**, 257–62.—A single-cyl diesel engine (max speed 1500 r.p.m.) with a swirl-chamber type of combustion chamber was found to cause excessive contamination of lub oil by combustion soot, despite the fact that the exhaust gas was not visible. The experiments described, using Pool gas oil as fuel, were made to discover the cause of this contamination.

When the injection timing was varied the change in smoke density, measured by a smoke meter, was independent of the change in specific fuel consumption, and minima occurred at 9 and 18 crank-angle degrees before top dead centre respectively. Thus the best combustion efficiency and the best thermal efficiency do not correspond. The period between the commencement of injection and combustion shows a discontinuity when ignition occurs as the piston reaches t.d.c. Exhaust analyses of CO (by reaction with I_2O_5), CO_2, and O_2 show that after the first 10° of injection advance there is no significant change in exhaust-gas composition.

The initial injection pressure was then varied, and it was found that whereas the specific fuel consumption was almost constant, the smoke density passed through a pronounced minimum.

Different spray nozzles were found to have little effect on specific fuel consumption; but had a considerable effect on smoke density.

It is concluded that contamination of lub oil by combustion soot is due to the poor combustion efficiency of a very small part of the fuel. This may be caused by droplets undergoing thermal decomposition rather than burning. H. C. E.

504. New Dutch medium-speed two-stroke diesel engine. Anon. *Gas Oil Pwr*, 1950, **45**, 233.—This Bolnes Type L engine is a two-stroke unit with cylinders of bore 190 mm and stroke 350 mm, and develops 50 b.h.p/cyl. A four-cyl prototype developing 200 b.h.p. at 430 r.p.m. has been tested at Delft Technical University. The fuel consumption in lb/b.h.p.hr. varies from 0·387 at 120 b.h.p. through 0·369 at 180 b.h.p. to 0·385 at 240 b.h.p.

Bosch fuel pumps inject the fuel into open-type combustion chambers in the piston crown. Pressure lubrication is supplied to the main bearings by a gear-type pump. Oil passes from the crosshead pins through holes in the piston-rods to cool the pistons internally. Lubrication of the crosshead piston and liner is supplied from the hollow piston pin. The water-cooling system operates on a closed circuit. H. C. E.

505. Gardner horizontal [diesel] engine. Anon. *Gas Oil Pwr*, 1950, **45**, 217–19.—This 6HLW type six-cylinder engine has cyl of bore 4¼ in and stroke 6 in, giving a swept volume of 511 cu. in. At full power it develops 112 b.h.p. at 1700 r.p.m. Over the range 1100–1500 r.p.m. the full-load torque and b.m.e.p. are at their max of 358 lb-ft and 106 p.s.i. respectively. Fuel consumption at full load and speed is 0·363 lb/b.h.p.hr.

Details of the engine construction are given. The pistons are of Al alloy with a deep, open combustion chamber in the crown, and carry three pressure and one scraper ring.

Four gal of lubricant is contained in an Al-alloy sump, and is distributed under pressure to the main bearings. The fuel is delivered through Gardner injectors by two C.A.V. pumps arranged horizontally. The other components are specially constructed for use with a horizontal engine. H. C. E.

506. Engineering details of the G.M. 110 diesel engine. Anon. *Auto Ind.*, 15.7.50, **103** (2), 38–9.—Details are presented of the General Motors 110 diesel engine, available for marine or industrial use. Specification details include: six cyl; bore 5 in; stroke 5·6 in; displacement 600 cu. in.; compression ratio 18 : 1; rated b.h.p. basic engine 275 at 1800 r.p.m., b.m.e.p. continuous rating 70 at 1600 r.p.m., weight 2960 lb. One of major design features is a new and more efficient gear-driven centrifugal blower, mounted at the rear above the flywheel. J. G. H.

507. Meadows new 4D330 diesel engine. Anon. *Gas Oil Pwr*, 1950, **45**, 250–2.—This direct-injection four-cyl model has cyls of bore and stroke 120 mm, giving a swept volume of 5·43 litres. At a max speed of 2200 r.p.m. the unit develops 80 b.h.p.,

and the max torque of 230 lb-ft at 1400 r.p.m. corresponds to a b.m.e.p. of 105 p.s.i. The compression ratio is 16 : 1.

The Al-alloy pistons carry three compression and two scraper rings, one of which is below the gudgeon-pin. The injection pump and governor gear are located in a separate assembly. The fuel-pump drive gear incorporates a governor which advances the point of injection as the engine speed increases. The injection pump supplies fuel to four-hole injectors at 175 p.s.i. H. C. E.

508. Meadows 970-type pressure-charged engine. Anon. *Gas Oil Pwr*, 1950, **45**, 166.—This engine is the pressure-charged form of a unit previously described (Abs. No. 1741, 1949), and develops 250 b.h.p. at 1650 r.p.m. Details of the engine construction are given. An exhaust-gas turbo-charger fitted at the rear end of the engine is water-cooled from the engine system.

The fuel consumption of this unit is 0·362–0·400 lb/b.h.p.hr. over the full load/speed range. The dry weight of the engine is 3900 lb. H. C. E.

509. New Vee-form M.A.N. diesel. Anon. *Gas Oil Pwr*, 1950, **45**, 151.—This engine is a four-stroke twelve-cyl 60° V-form unit with cyl bore and stroke of 175 and 210 mm respectively. Naturally aspirated or pressure charged, it develops 550 or 800 b.h.p.

Brief constructional details are given.

Lub oil is circulated by a gear-type pump through an external cooler. The camshaft and main bearings are pressure lubricated, and the pistons are splash lubricated. Engine cooling water is circulated by means of a centrifugal pump. H. C. E.

510. David Brown marine engine. Anon. *Gas Oil Pwr*, 1950, **45**, 182.—The diesel unit is a four-cyl four-stroke engine with cyls of bore and stroke 3½ in and 4 in. The C.R. is 15·8 : 1, and the max output is 31 b.h.p. at 1800 r.p.m.

The engine design and construction are briefly described.

A C.A.V. injection pump supplies fuel to four-hole injectors. The main bearings, crankpins, valve camshaft, and rockers are pressure lubricated by means of a submerged gear-type pump. A 12-V electric motor is provided for starting.

The petrol/paraffin unit has cyl dimensions of 3½ in and 4 in, and operates at a compression ratio of 4·7 : 1 to give a max output of 32 b.h.p. at 1800 r.p.m. The basic design is similar to that of the diesel engine. H. C. E.

511. Evaporative cooling for marine internal combustion engines. J. F. Alcock. *J. Inst. Mar. Engrs*, Oct. 1950, **62** (10), 327.—Evaporative or steam cooling has not yet come into general use in spite of the consideration given to it for some considerable time. To be adopted steam cooling must obviously show a balance of advantage over water cooling, which is at present universal in marine service. A comparison between the two systems is made, and the disadvantages of the typical water-cooled system described, particular reference being made to temp control, simplicity and cost, cooling of hot spots, dissipation of heat. Design problems are then described, and the general conclusions are that the most promising field for steam cooling seems to be in the moderately rated engines of medium or fairly large size. In this range steam-cooling has slight advantages, particularly as regards temp control. For really severe conditions fast water-cooling seems to be better but more expensive. Individual designs will need a thorough examination as regards water and steam flow areas, blanketing risks, etc. Information on conditions prevailing at hot spots is deplorably scanty, and research on this subject would be of value to both steam- and water-cooling. A. S.

512. Canadian diesel-electric locomotive. Anon. *Gas Oil Pwr*, 1950, **45**, 154–6.—This unit is powered by a 1500-b.h.p. turbo super-charged V-12 engine which drives four electric motors giving a starting tractive force of 62,500 lb at 25% adhesion. It carries 160 gal lub oil, 960 gal fuel, and 200 gal cooling water.

The Alco diesel engine has twelve cyls each of bore and stroke 9 in and 10½ in respectively. It is a four-stroke engine with two inlet and two exhaust valves per cyl.

A brief description of the electrical equipment and control system is given. H. C. E.

513. Diesel-mechanical locomotive for the Southern Region. Anon. *Gas Oil Pwr*, 1950, **45**, 177.—This 0-6-0 locomotive is powered by a Paxman 12 RPH V-type diesel engine developing 500 b.h.p. at 1250 r.p.m. The cyls are of dimensions 7-in bore and 7¾-in stroke. Between the cyl banks are situated the fuel pumps, arranged in two units and driven by the camshaft, which is located in a separate housing and driven by a gear train from the free end of the engine.

Power is transmitted through a fluid coupling and a gearbox which provides for speeds from 5 to 36 m.p.h., giving a tractive effort of from 33,500 to 4600 lb.

Details of the auxiliary equipment are given. H. C. E.

514. Impressive performance by big diesel locomotive. Anon. *Gas Oil Pwr*, 1950, **45**, 232.—This locomotive is powered by two pressure-charged Sulzer 2200-b.h.p. engines, each with twelve cyls of bore and stroke 12·2 and 15·4 in, running at 700 r.p.m. After completing 1,000,000 km in service it has been refitted and measurements have been made of the working parts. The extent of wear of the various components is given in detail.

During the period of service the fuel and lub oil consumptions averaged 13·4 and 0·30 lb/mile respectively. H. C. E.

515. Milestones in Sulzer railway engine development. Anon. *Gas Oil Pwr*, 1950, **45**, 253–5.—A short description of typical diesel railway engines manufactured by Sulzer Bros from 1912 to the present day. H. C. E.

516. Mixed traffic diesel locomotive for Eire. Anon. *Gas Oil Pwr*, 1950, **45**, 226.—This locomotive is powered by a Sulzer 6LDA28 type engine with cylinders of 11 in and 14½ in bore and stroke. The 1-hr and continuous ratings are 915 and 815 b.h.p. at 750 and 700 r.p.m., corresponding to a b.m.p. of 115 and 110 p.s.i. Specific fuel consumption is 162 gram/b.h.p.hr. on continuous rating.

The electrical equipment comprises main and auxiliary generators, four force-ventilated traction motors, and the Sulzer form of regulator control.

A speed of 17½ m.p.h. was maintained up a grade of 1 in 84 while hauling a 360-ton freight train; and 58 m.p.h. up a grade of 1 in 1143 when hauling a 120–130-ton passenger train. H. C. E.

517. Design analysis of free-piston engines. A. K. Oppenheim and A. L. London. *Auto Ind.*, 1.7.50, **103** (1), 46–50, 76–8.—The applications of free-piston engines to air-compressors and to compounded-gas turbines are noted, and details are presented of research carried out at stanford University on the design of free-piston engines for use with gas turbines. The design data so obtained were applied to a Pescara gas generator, and the experience resulting was used as a basis for certain modifications in design. J. G. H.

518. Huey gas turbines. C. C. Willis and E. C. Goldsworth. *Mech. Engng*, Nov. 1950, **72** (11), 881.—The paper describes the engineering and construction problems involved in the application of a 3500-kW simple-cycle, locomotive-type, gas-turbine-generator unit to an existing electric-utility steam plant. At the end of the war the Huey station of the Oklahoma Gas and Electric Company had a total boiler capacity equivalent to 51,000 kW and turbine capacity of 56,000 kW. Several proposals were considered for making up the deficiency in steam-generating capacity and for making available the additional 5000 kW from the turbines. It was eventually decided to instal the above turbine equipped with an air pre-cooler and a gas heat-exchanger for heating the steam-plant feedwater. It was shown that this would result in a 7000-kW gain in plant capability—4000 from the gas-turbine-driven generator unit, and 3000 from the release of extracted steam for generation instead of feed-water heating. The paper describes the general arrangements and connexions to existing station, accessory equipment such as air precooler, gas-fuel compressor, feed-water control, and air and gas ducts. Details are also given of the changes necessary to adapt the turbine for stationary electric utility operation. A. S.

519. Electrical equipment. Anon. *Auto Engr.*, Nov. 1950, **40** (533), 356.—A very high standard has been attained both as regards reliability and also ease of servicing

of the basic units employed for starting and lighting installations. This question is bound up with standardization, and it would seem that it is only in this direction that progress can be expected. The paper deals with the important design features of starting motors (C.A.V., Simms, and Lucas types); batteries (Young, Peto and Radford, Lucas, Nife); dynamos (Simms, C.A.V.); lamps (C.A.V.); direction indicators. A number of photographs and drawings are given showing the design features and general arrangement of the above electrical equipment. A. S.

Safety Precautions

520. Crankcase explosions. F. V. Cook. *Oil Gas J.*, 16.11.50, **49** (28), 291.—Some crankcase explosions with diesel and dual-fuel engines are discussed, and methods of prevention given. The author considers fallacious the general assumption that oil and air mixture in an engine crankcase is normally too rich to burn and that piston blow-by consists primarily of the products of combustion. He suggests that explosions result from ignited mixtures of vapour and air that fill spaces between droplets of oily mist in crankcases, and that piston blow-by is negligible.

Of the preventive methods discussed, that involving use of low-pressure explosion valves is considered most satisfactory by the author. G. A. C.

521. Marine gassing and fire and explosion hazards. A. S. Minton. *Trans. Inst. mar. Engrs*, Oct. 1950, **62** (10), 307.—Marine fires can be divided into the four following types: (*a*) explosions and resultant fires; (*b*) oil or petrol fires; (*c*) dry-dock fires; (*d*) passenger-liner fires. Various plates are included in the paper showing examples of these. Legislation and recommendations are given relating to: (i) the ship, its condition and equipment; (ii) the cargo, duties and liabilities of shipowner and cargo-owner; (iii) duties of the ship in harbour while carrying explosives, petroleum, or coal; (iv) regulations and requirements in connexion with the repair of ships. Methods of dealing with marine gassing, fire, and explosions hazards are described under these three main categories. Attention is given to the various types of fire extincteurs and their practical application, and to the different types of fire encountered. Marine explosions can be due to petroleum, high explosives, dust explosions, chemicals, bursting of steam boilers. Gassing hazards are described under the following headings: (i) vapour from cargo of inflammable liquids; (ii) carbon dioxide accumulation; (iii) fumigation; (iv) refrigerating gases; (v) carbon monoxide poisoning, and (vi) nitrous fumes. A comprehensive table is given showing the properties of a wide range of gases and indicating their influence on human reactions. The relative advantages of gas-masks, nose masks, and oxygen apparatus for dealing with petroleum vapours and hydrogen sulphide are also tabulated. A. S.

522. Emergency electrical equipment justified as initial plant investment. J. M. Crothers. *Refiner*, 1950, **29** (9), 133–4.—In cases where frequent power failures of long duration to important process units are normal, it is justifiable to maintain auxiliary electrical supply. Infrequent power failures of short durations, not causing complete shut-downs or long periods of return to full on-stream operations, will not justify such an expenditure. Direct-current controls are being superseded by alternating-current reactor and capacitor devices, thus banks of batteries are no longer suitable. A description of a recent installation requiring three-phase and single-phase emergency power is given. In this, a small turbo generator is kept in continuous slow motion and is speeded up automatically on power failures by the opening of the main steam-valve controlled by a solenoid. R. W. G.

523. A striking demonstration. Anon. *Paint Technol.*, Nov. 1950, **25** (179), 484.—The "'Pyrene' P.D. 20 CO_2 Dry Chemical Fire Extinguisher" is described. A dry chemical is expelled by carbon dioxide, the latter not only acting as a powerful expellent for the chemical but also as a supplementary fire-fighting agent. On the ground the mixture looks like dry snow, and on top of residual solvent after a fire has been extinguished it has the appearance of a compact scum. The main container is of "Parkerized" solid drawn steel, and can withstand a pressure of 600 p.s.i. A flexible delivery hose is attached to the container, and it incorporates a valve which enables

the operator to shut off and restart the discharge at will. With this appliance the extinguisher chemical can easily be driven a distance of 15 ft and anyone can use it. Fires up to 8 or 10 sq. ft. in area can be stopped in less than 2 sec with this extinguisher of 2 gal capacity and containing 20 lb of the dry chemical. It is claimed that the extinguisher can put out a fire 8 sq. ft. in area six times with one charge. Tests were made on fires of petrol, acetone, butanol, di-*iso*butylene, *iso*propyl alcohol, and toluol. Fires of these substances and other larger fires of petrol and petrol on fuel oil were all extinguished in a few seconds. D. K.

MISCELLANEOUS

524. Advances in petroleum refining. W. M. Holaday. *Refiner*, 1950, **29** (9), 77–92.—A review of the general trends in the development of petroleum refining since its inception. The article is chiefly concerned with the progress which has taken place in the U.S., since this has been the most extensive owing to the high U.S. petroleum consumption. Petroleum-products consumption for the U.S. is reported to be 12 brl per person per year, while in Europe (excluding U.S.S.R.) it is 0·65 brl per person per year. The motive behind past and current development in the U.S. is the demand for more and higher O.N. gasolines and distillate fuels in general. This has resulted in a change from 100% of distillate fuels in 1914 being straight run to only 25% in 1950, the remainder being produced by cracking (65% cat and 10% thermal). The U.S. consumption of petroleum products for 1950 is estimated at 6,000,000 b.d. Several diagrams representing the growing and changing importance of the cracking processes, etc., are given, both for the U.S. and other areas.

Of the various petroleum processes, the following are specifically considered with reference to increased gasoline yield and quality: thermal cracking and reforming; catalytic cracking; polymerization; alkylation and isomerization; polyforming and hydroforming; gasoline treating (stability improvement and sulphur conversion or removal). Sections on trends in motor gasoline utilization and future processing techniques are also given. It is suggested that improved petrol-engine design be favoured in preference to high-O.N. gasoline demand. R. W. G.

525. Current trends in petroleum refining. F. H. Garner and F. Morton. *J. Inst. Fuel*, 1950, **23** (134), 297.—A table showing the comparative situation of U.K. refineries in 1948 and in 1952–53 is given.

The refining industry of the U.K., which previously had a capacity of 100,000 b.d. of a very specialized nature, is to be expanded so that by 1953 its capacity will be 400,000 b.d. It is anticipated that about 65% of Europe's crude oil requirements under the expanded programme will be met by crudes from the Middle East. These crudes, in general, yield fractions of high wax content, but residues are asphaltic and have a high sulphur content. During the last three years the American refining industry has greatly expanded. The rise in demand for middle distillates combined with increased demand for gasoline has been the subject of many studies. The development of catalytic cracking has enabled this demand to be met. Diagrams showing original, wartime, and recent designs of catalytic cracking units are presented and discussed. Coking processes are briefly considered, and it is thought that if the proposed expansion of the U.K. refining industry is to be economic a satisfactory continuous coking process would be a major contribution to this end. Catalytic reforming has been developed, and among the newer processes "platforming." Other factors which may affect the overall realization per barrel of crude oil are the efficient utilization of gases, obtained in cracking operations, the production of high-priced fractions, and the recovery and utilization of sulphur and nitrogen compounds present in the crude oil. The hypersorption process for separation of gas streams by adsorption on a moving bed of charcoal is described.

The use of the Koch Kaskade column in the distillation and sulphur removal processes is also discussed. Several tables of data and forty-four literature references are included. D. K.

526. Heavy demand boosts California refining. Anon. *Oil Gas J.*, 9.11.50, **49** (27), A46.—A review is given of increased demand for petroleum products, and of projects under way to meet demand in the Pacific Coast states.

Gasoline consumption has almost doubled since 1939, diesel oil demand has increased by 137%, and heavy fuel oil by 37%.

Plants to process heavy crudes and residuals into gasoline and distillates are being constructed or planned at Richmond, Torrance, and Oleum, Calif.

Total crude oil capacity of Californian refineries is now 1,044,800 b.d. G. A. C.

527. 50 years of progress in the French petroleum industry. H. Weiss. *Bull. Assoc. franç. Tech. Pétrole*, 1950 (84), 7–21.—A review, confined to the refining side of the industry, which in France can be divided into three periods, pre-1914, 1924–40, 1946 onwards. A refinery at Rouen was, in the pre-1914 period, the first to use the Edeleanu process on a commercial scale. Modern refining processes are briefly outlined.

V. B.

528. Venezuela—oil country of contrasts. J. de Coudier. *Bull. Assoc. franç. Tech. Pétrole*, 1950 (84), 23–44.—A lecture, describing the growth and extent of the Venezuelan petroleum industry, together with some account of the country and conditions therein. Some past and current oil statistics are quoted. V. B.

529. What's the present picture on oil in the Far East? W. E. V. Abraham. *Oil Gas J.*, 16.11.50, **49** (28), 226.—Data and speculative opinion are given on the existing situation and possible future in all countries which lie east of the Urals and the Persian Gulf and west of the Americas—including Asiatic Russia, India, Pakistan, Indonesia, Japan, and Australasia.

History and results of exploration, transportation, consumption, and refining situation are also dealt with, together with probable future relationship between production and consumption. G. A. C.

530. SO_3 and SO_2 in exhaust gases of internal-combustion engines. M. J. van der Zijden, J. E. van Hinte, and J. C. van den Ende. *J. Inst. Petrol.*, 1950, **36**, 561–73.—The SO_3-content of the exhaust gases of some spark-ignition and diesel engines was determined in order to study the influence of SO_3 on wear and fouling of internal-combustion engines.

The quantity of SO_3 present was found to be very small in all cases, by far the greater part of the sulphur being present as SO_2. The correct determination of SO_3 in addition to SO_2 in the exhaust gas is, however, very difficult, because SO_3 is rapidly oxidized during sampling and analysing, due to the presence of compounds of the peroxide type. Oxidation will also occur on cold cyl walls, and the SO_3 thus formed is considered as a main source of wear and fouling of engines. There is obviously little relation between the true SO_3-content of the exhaust gas and the SO_3 found on the cyl walls, and SO_3-determinations of the exhaust gas are therefore of little use for studying engine wear and fouling phenomena. A. R. W. B.

531. Petroleum in agriculture. F. W. Ellis. *I. P. Review*, 1950, **4**, 317.—Petroleum products are used in agriculture in two main ways. The first is in agricultural machinery dating back to 1856, when Fowler introduced his steam ploughing unit. In the U.K. to-day, there are 230,000 kerosine tractors, 50,000 petrol tractors, and 15,000 diesel tractors, using 225, 25, and 12 million gal/annum of their respective fuels. Thus, including an allowance for lubrication, the total consumption of petroleum products used in this way is valued at about £17½ million per annum. Diesel tractors are not so popular in the U.K. as elsewhere because of high manufacturing costs, but an increase in the number employed is expected in the near future. Artificial driers are coming into vogue with the increased use of the combined harvester, and about 6 million gal of fuel oil are being used. 200 sec fuel oil is becoming increasingly popular for this. Special products like detergent oils, greases, and rust-preventives are also used.

The second main use of petroleum products is as an agent in crop growing. Petroleum oils are used as insecticidal sprays, as weed-killers (especially selective weed-killers), and as synergists to increase the toxic effectiveness of other chemicals.

Aerial spraying and dusting of crops also has a future. R. H.

BOOK REVIEW

Oilfield Exploration and Development. Vol. I. Principles. Vol. II. Practice. A. Beeby-Thompson. London: The Technical Press Ltd., 1950. £4 4*s*. per set. Pp. lxv + 546 + 32 and lv + 630 + 32.

This new edition of Mr Beeby-Thompson's well-known treatise, first published in 1925, the author frankly admits to be a reprint with appendices.

Its essential value is, therefore, its clear descriptions of basic principles and practices over the vast front of the petroleum industry's activities from exploration through the intermediate stages of production and refining to transport and storage.

The new appendices describe the growth of world production and the extent to which revolutionary changes in technique have called for a high degree of specialization in every department of the industry.

The author contends that it is impossible to include in one treatise descriptions of all the advances in technique which have occurred, and he and his publishers have preferred to preserve the subject-matter of the original edition intact.

This has the result that some of the material, photographs, and figures are out of date, and much of it is inapplicable at the present time. This, of course, applies more to the "Practice" volume than to the one on "Principles," and for this reason it might have been preferable to rewrite at least the "Practice" volume.

E. C. S.

BOOKS RECEIVED

Fuel Research 1939 to 1946. Dept of Scientific and Industrial Research. London: H.M. Stationery Office, 1950. Pp. 69 + v. 3*s*. net.

The last report of the Fuel Research Board and of the Director of Fuel Research was for the year ended March 31, 1939. The reports contained in this volume cover the seven-year period to 1946 and give details of considerable importance to the fuel industries as a whole.

There is much to interest and be of value to the petroleum technologist, particularly in the chapters on the synthesis of hydrocarbons and on the production and treatment of hydrocarbon oils. The sub-headings of the first of these chapters are catalytic synthesis of methane, and the synthesis of hydrocarbons from CO and H, *i.e.*, the Fischer–Tropsch process. The chapter on hydrocarbon oils deals with the production of toluene, the sludging and corrosion in benzole-absorption plants, colloidal fuel, and treatment by silent electric discharge, *i.e.*, the "Elektrion" or "Voltol" process.

Other matters in the report which will be found of interest refer to the solidification of liquid fuels, work for the Petroleum Warfare Department, and ester waxes from lignite and peat.

References to 231 articles in the literature are appended.

Proceedings of Third Conference on Chemical Works Safety. London: Association of British Chemical Manufacturers, 1950. Pp. 184. 5*s*. net.

This Conference was held during the period September 29–October 1, 1950, and the report contains the papers presented and the discussion. The papers are of interest to the petroleum industry, and in addition to one on that industry in particular mainly deal with pressure vessels and cause of fire.

Report of the Government Chemist for the Year Ended 31st March, 1950. London: H.M.S.O., 1950. Pp. 35. 1*s*. net.

Horizontal Diesel Engines. R. A. Collacott. London: Temple Press Ltd., 1950. Pp. 122 + vii. 8*s*. 6*d*. net.

In this book Dr Collacott confines his subject to the simple horizontal diesel engine of up to 300 b.h.p., as used mainly in industrial establishments. The various chapters deal with principles of operation and details of engine, the supply of fuel, coiling and lubrication, etc., with information on some British engines. Inspection and diagnosis charts are included.

Surface Active Agents. A. M. Schwartz and J. W. Perry. New York: Interscience Publishers, 1949. Pp. xi + 579.

Mining Laws of Canada. A. Buisson. Ottawa, Canada: Department of Mines, 1950. Pp. ix + 133. 50 cents.

The fourth edition of a digest of Dominion and provincial laws and regulations.

Canadian Mineral Industry in 1948. Ottawa, Canada: Department of Mines, 1950. Pp. vi + 126. 25 cents.

Reports by the Staff of the Canadian Mines Branch of developments during 1948 of twenty-four metals, thirty-six industrial minerals, and five fuels—coal, coke, natural gas, peat, and crude petroleum.

B.S. 673: 1959 Pneumatic Tools and Accessories. London: British Standard Institution, 1950. Pp. 25. 3*s*. net.

B.S. 1501–6: 1950. Steels for Use in the Chemical, Petroleum and Allied Industries. London: British Standards Institution, 1950. Pp. 66. 12*s*. 6*d*. net.

This is part of a comprehensive scheme of codes for pressure vessels, and covers plates, bars and sections, forgings, castings, and bars for bolting material.

B.S. 1310: 1950. Coal Tar Pitches for Building Purposes. London: British Standards Institution, 1950. Pp. 22. 3*s*., post free.

B.S. 1644: 1950. Dimensions of Unscreened Camshaft Speed Magnetos. London: British Standards Institution, 1950. Pp. 18. 2*s*. 6*d*., post free.

B.S. 1677: 1950. Draughtsmen's Drawing Pins. London: British Standards Institution. Pp. 6. 1*s*., post free.

B.S. 1649: 1959. Guards for Shaft Couplings. London: British Standards Institution. Pp. 12. 2*s*., post free.

B.S. 1600–7: 1950. Wrought Steel Pipe for the Petroleum Industry. London: British Standards Institution. 1950. Pp. 42. 10*s*. 6*d*. net.

This is part of a series to deal with petroleum industry equipment.

B.S. 1658: 1950. Housings for Hydraulic Seals. London: British Standards Institution, 1950. Pp. 11. 2*s*. net.

B.S. 1686: 1950. Long-Period, High Sensitivity Tensile Creep Testing. London: British Standards Institution. Pp. 9. 2*s*., post free.

B.S. 1687: 1950. Medium-Sensitivity Tensile Creep Testing. London: British Standards Institution. Pp. 9. 2*s*., post free.

B.S. 1688: 1950. Determination of Time to Rupture Under Stress. London: British Standards Institution. Pp. 9. 2*s*., post free.

B.S. 1689: 1950. Fire Buckets. London: British Standards Institution. Pp. 8. 2*s*., post free.

B.S. 594: 1950. Rolled Asphalt, Asphaltic Bitumen, and Fluxed Lake Asphalt. (Hot Process.) London: British Standards Institution. Pp. 55. 6*s*., post free.

B.S. 903: 1950. Methods of Testing Vulcanized Rubber. London: British Standards Institution. Pp. 247. 21*s*., post free.

Report of the Geological Survey Board for the Year 1949. London: H.M.S.O., 1950. Pp. 18. 9*d*. net.

APPLICATIONS FOR MEMBERSHIP OR TRANSFER

FEBRUARY 1951

The following have applied for admission or transfer to the Institute. In accordance with the By-laws, the proposals will not be considered until the lapse of at least one month after the publication of this *Journal*, during which time any Fellow, Member, or Associate Member may communicate by letter to the Secretary, for the confidential information of the Council, any particulars he may possess respecting the qualifications or suitability of the candidate.

The object of this information is to assist the Council in grading the candidate according to the class of membership.

The names of candidates' proposers and seconders are given in parentheses.

Applications for Membership

EGBEARE, Thomas Henry Elliott, chemist, Anglo-American Oil Co. Ltd. (*E. J. Boorman; E. H. Wild.*)

HODGE, Harold Percy, assistant chemist, Anglo-American Oil Co. Ltd. (*E. J. Boorman; E. H. Wild.*)

PALMQUIST, Fredrik Teodor Emanuel, research chemist, A. B. Separator-Nobel. (*F. Sager; R. H. Dolton.*)

RAINE, Joseph Curry, plant superintendent, British Petroleum Chemicals Ltd. (*D. N. McKinlay; W. L. Wood.*)

SHAPLAND, John Dee, technical assistant to managing director, Foamite Ltd. (*E. B. Evans; D. M. Newitt.*)

SHORTEN, George Alfred, general manager, Trade Lubricants (Clifton) Ltd. (*A. Dunbar; G. Sell.*)

SWAIN, Arthur, supervisor (Industrial and Automotive Lubricants), Shell-Mex & B.P. Ltd. (*F. Green; W. C. Timothy.*)

TUEMMLER, Frederick Donald, head analyst, Shell Development Co. Ltd. (*H. Hyams; G. W. Dorrell.*)

WILLIAMS, Tudor Vaughan, senior development engineer, National Oil Refineries Ltd. (*H. Murphy; E. J. Horley.*)

Transfers

BROOKBANK, John Anthony, technical assistant, "Shell" Refining & Marketing Co. Ltd. (*H. E. F. Pracy; E. Pay.*) (*Student to Associate Fellow.*)

BURGESS, Bruce Henry, research physicist, Esso Development Co. Ltd. (*E. B. Evans; H. L. West.*) (*Student to Associate Fellow.*)

LESTER, Leslie John, chemist, Messrs. Arthur Brown & Co. Ltd. (*F. A. Ostler; R. W. Sheen.*) (*Student to Associate Member.*)

WATSON, Kenneth James, oil technologist, Attock Oil Co. Ltd. (*Student to Associate Member.*)

NEW MEMBERS

The following elections have been made by the Council in accordance with the By-laws.

Elections are subject to confirmation in accordance with the By-laws.

As Fellows

DE LA COVA, C. P. DIMITRU, C. A.

Transferred from Member to Fellow

SNOW, P. H.

As Member

HOLMES, J. B.

As Associate Fellows

BURGESS, H. G.
BURJORJEE, H. R.
DANIELS, S. G.
FREEBORN, A. S.
HALL, R. T. W.
JENKINS, T. E.
MILES, P. F.
NICOLL, H. G.

Transferred from Associate Member to Associate Fellow

COBLEY, R. C.

Transferred from Students to Associate Fellows

JONES, M. T. B.
MORRIS, D. M.

As Associate Members

CROWLEY, J.
MACKIAN, J.
STEVENSON, S.

As Students

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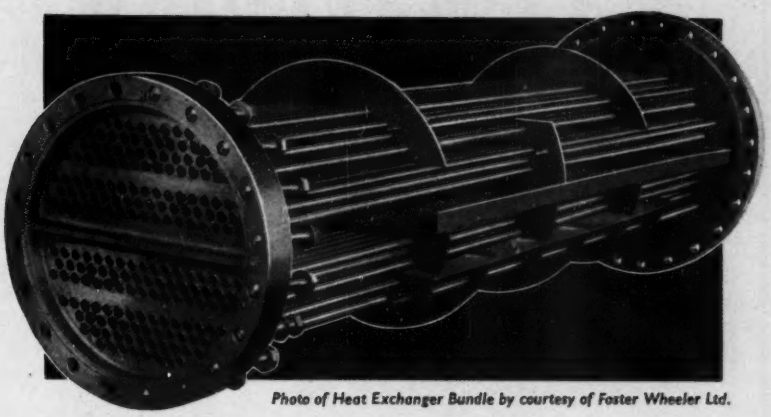

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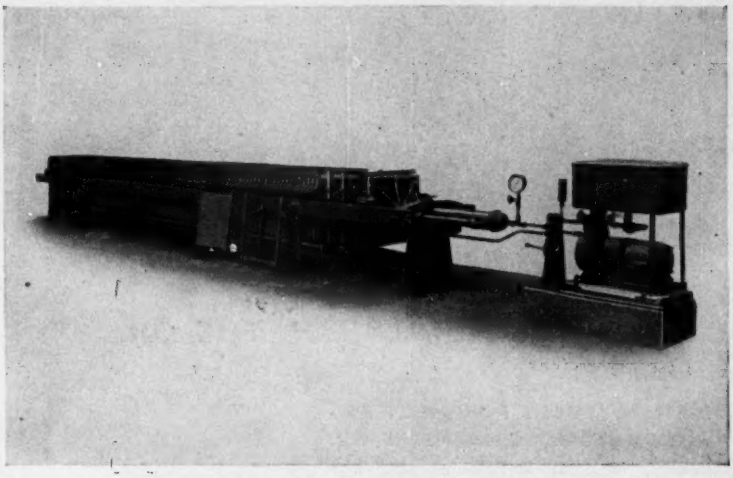

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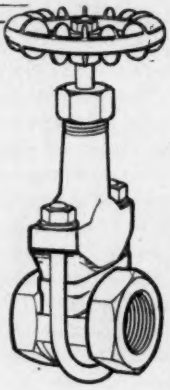

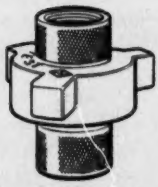

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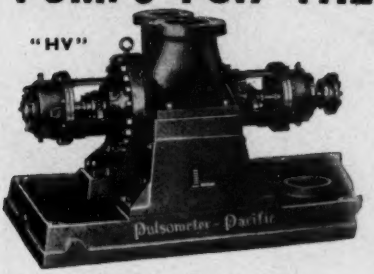

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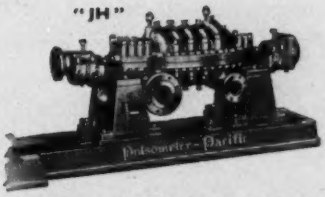

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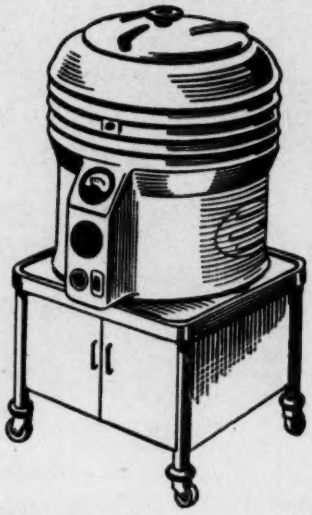

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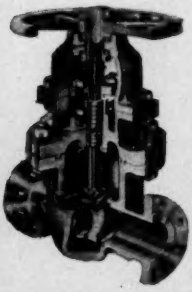

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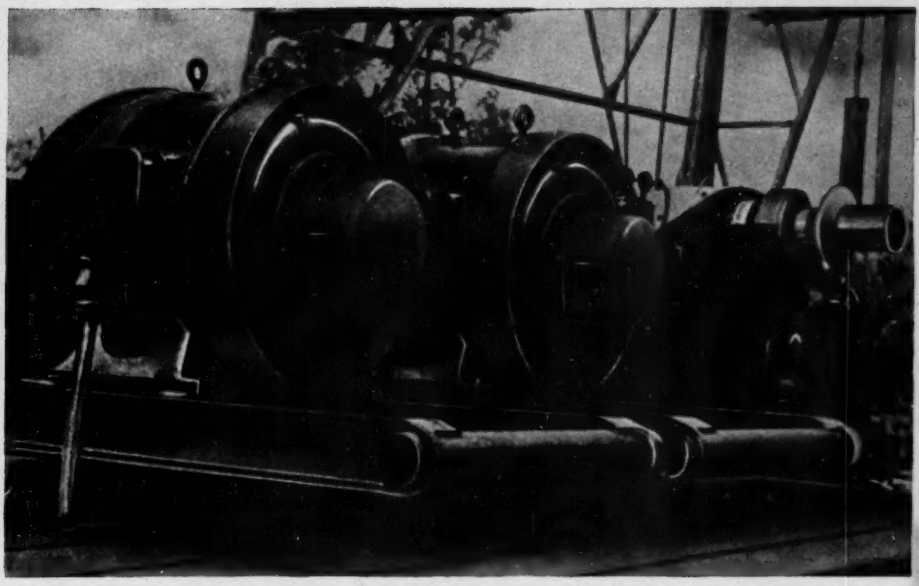

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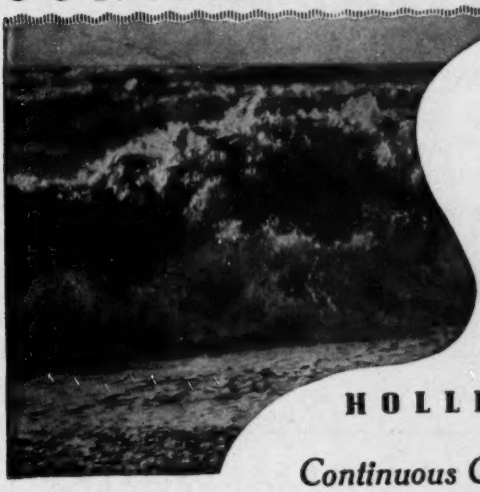

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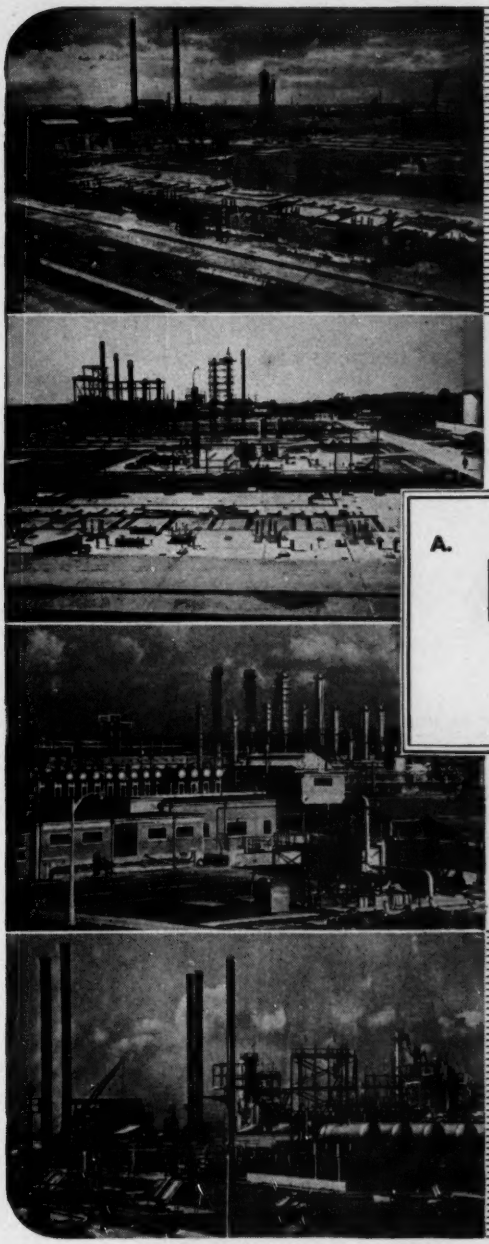

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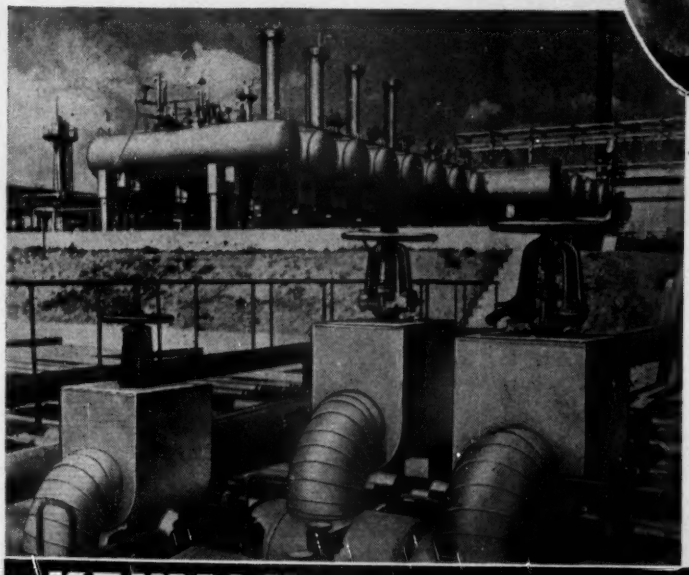

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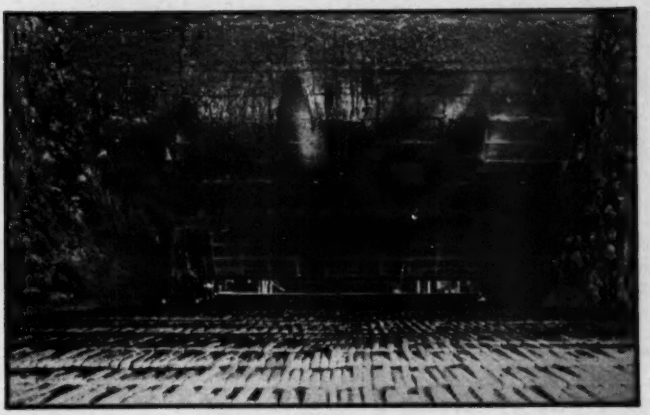

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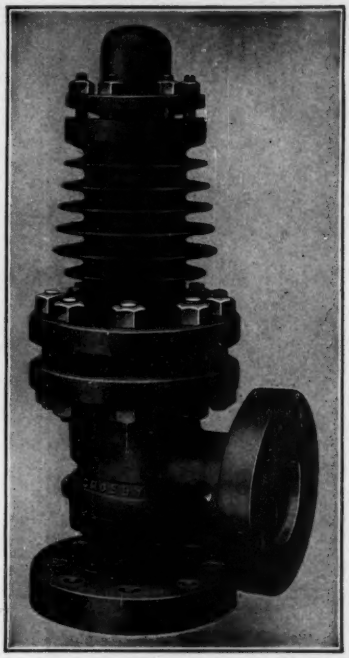

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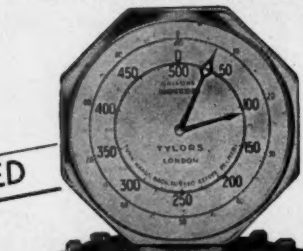

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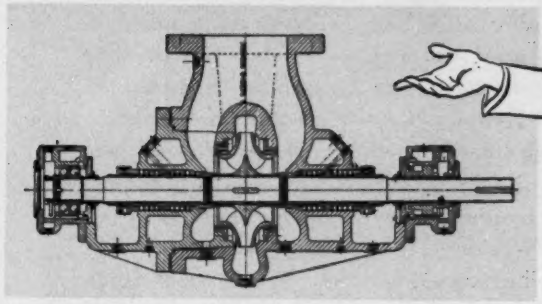

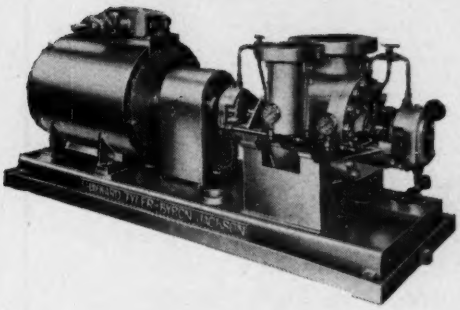

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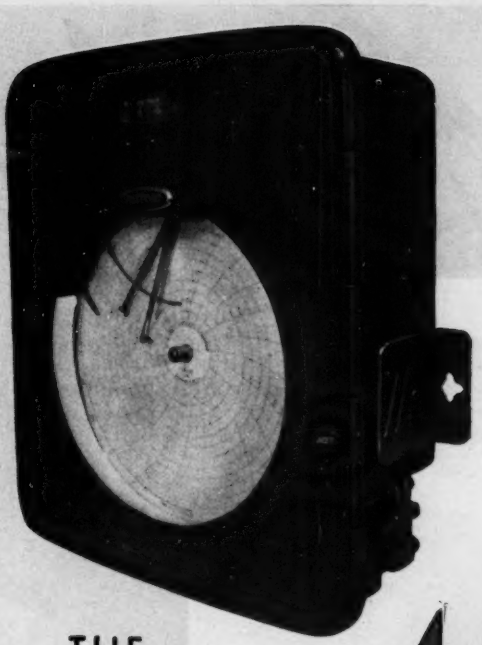

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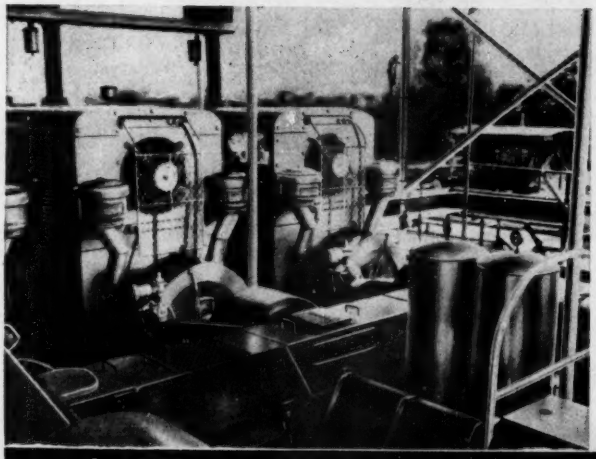

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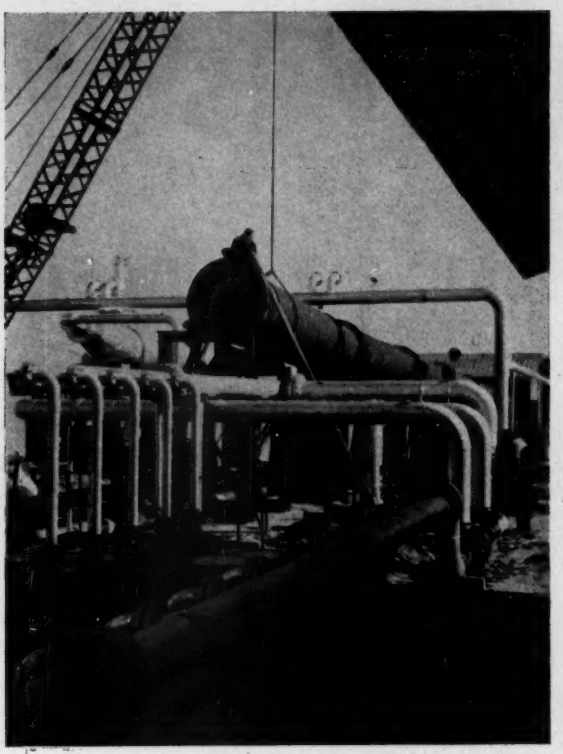

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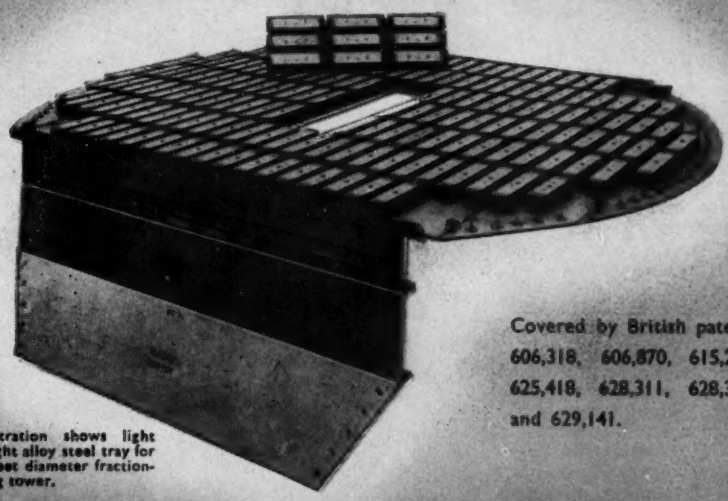

Illustration shows light weight alloy steel tray for 12 feet diameter fractionating tower.

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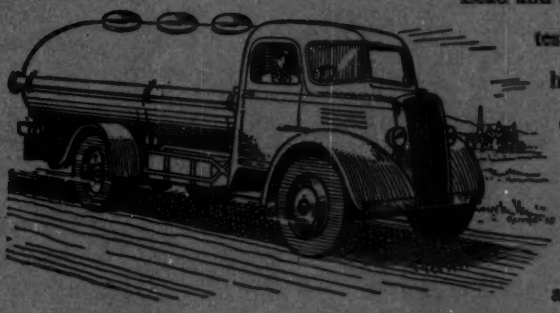

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